

Joint
Transportation
Research
Program

JTRP

FHWA/IN/JTRP-98/8

Final Report

**FIELD DEMONSTRATION OF HIGHWAY
EMBANKMENT CONSTRUCTED USING WASTE
FOUNDRY SAND**

**Barry K. Partridge
James E. Alleman**

September 1998

**Indiana
Department
of Transportation**

**Purdue
University**

FINAL REPORT

FIELD DEMONSTRATION OF HIGHWAY EMBANKMENT CONSTRUCTED USING WASTE FOUNDRY SAND

FHWA/IN/JTRP – 98/8

by

**Barry K. Partridge, *PhD. Student*
Dr. James E. Alleman, *Professor***

**Joint Transportation Research Program
Project No: 36-36Z
File No: 6-14-26**

Prepared as part of an Investigation Conducted by the

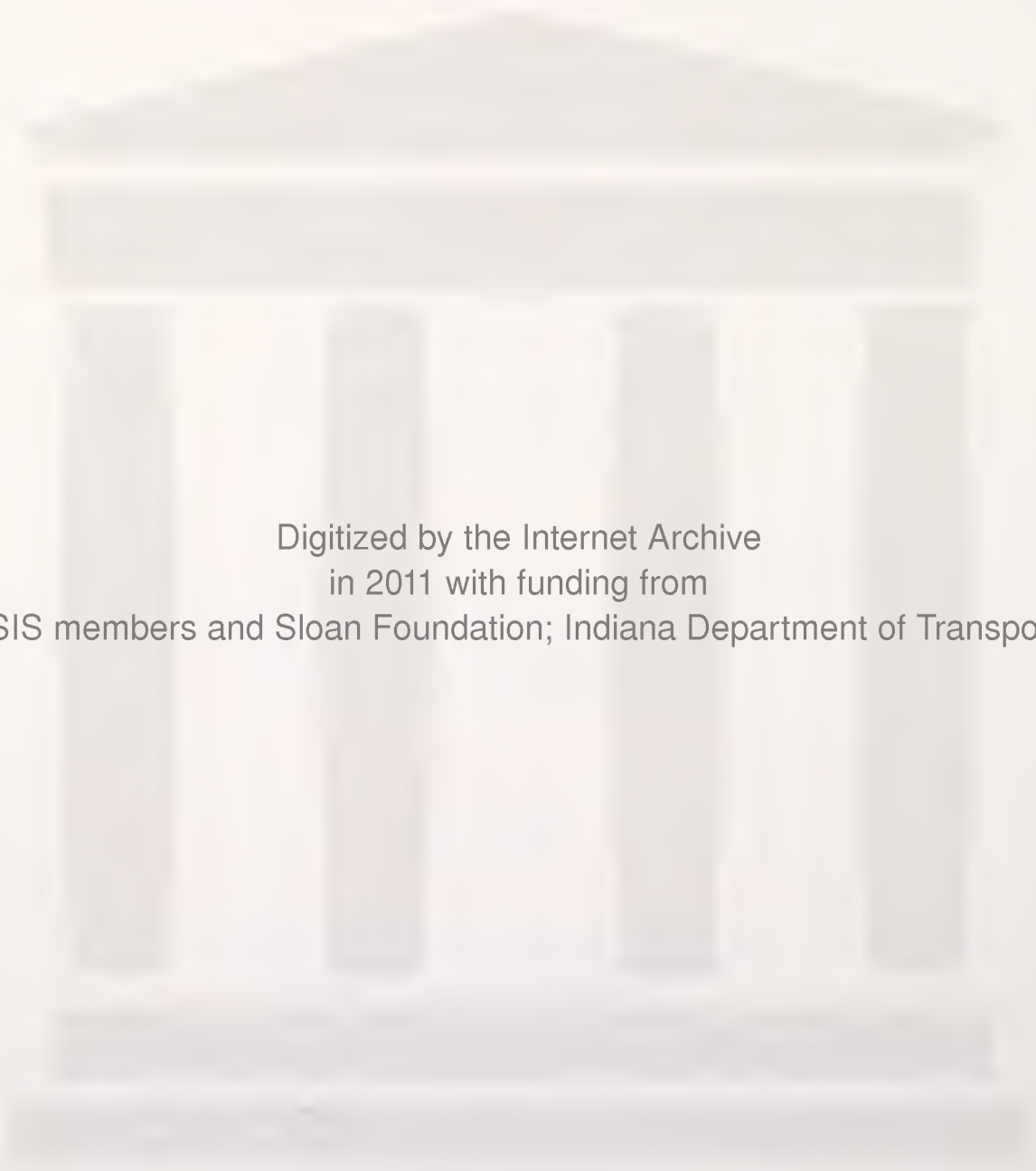
**Joint Transportation Research Program
Purdue University**

funded by the

**Indiana Department of Transportation and
Federal Highway Administration**

The contents of this report reflect the views of the authors who are responsible for the facts and accuracy of the data presented herein. The contents do not necessarily reflect the official views of, or policies of any of the sponsors. Furthermore, this report does not constitute a standard, specification, or regulation.

**School of Civil Engineering
Purdue University
West Lafayette, IN 47907-1284**



Digitized by the Internet Archive
in 2011 with funding from
LYRASIS members and Sloan Foundation; Indiana Department of Transportation

TECHNICAL REPORT STANDARD TITLE PAGE

1. Report No. FHWA/IN/JTRP-98/8	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle Field Demonstration of Highway Embankment Constructed Using Waste Foundry Sand		5. Report Date September 25, 1998	
		6. Performing Organization Code	
7. Author(s) Barry K. Partridge and J. E. Alleman		8. Performing Organization Report No. FHWA/IN/JTRP-98/8	
9. Performing Organization Name and Address Joint Transportation Research Program Purdue University West Lafayette, Indiana 47907-1284		10. Work Unit No.	
		11. Contract or Grant No. SPR-2136	
12. Sponsoring Agency Name and Address Indiana Department of Transportation State Office Building 100 North Senate Avenue Indianapolis, IN 46204		13. Type of Report and Period Covered Final Report	
		14. Sponsoring Agency Code	
15. Supplementary Notes Prepared in cooperation with the Indiana Department of Highways and Federal Highway Administration. This project was funded in part by the FHWA Priorities Technology Program.			
16. Abstract Over 9 million Mg of waste foundry sands (WFS) are produced annually in the United States as a by-product of the metal casting industry. The majority of WFS are deposited in restricted or sanitary waste landfills. Considerable savings is available to the metal casting industry through the development of reuse applications for their WFS and generators are often willing to provide WFS to a job site at no cost to the end user. Laboratory investigations have indicated that WFS from ferrous foundries can provide the necessary engineering properties for a highway embankment and that the Microtox™ bioassay test can be used to screen the 'toxicity' of WFS to prevent a negative environmental impact. In 1996, the Indiana Department of Transportation (INDOT) and Purdue University constructed a demonstration embankment using WFS. WFS and control embankments were instrumented to monitor geotechnical and environmental performance. Stockpile and job site 'grab' WFS samples were also tested. Detailed geotechnical results are presented in a companion report by Fox and Mast (1998). In general, results indicate that WFS can perform well as a structural fill with strength and deformation characteristics comparable to natural sand, but cannot be considered as freely draining. Environmental testing consisted of Microtox™ and Nitrotox bioassays, ion chromatography, and inductively coupled plasma testing for metals. Bioassay results indicate the WFS have not resulted in inhibitions (toxicity) higher than those expected from natural sands. Ion migration from the WFS into the foundry sand lysimeter was found, supporting bioassay data, but at concentrations below reuse regulatory criteria. Metal concentrations were generally below Indiana regulatory reuse Type III criteria and typically below Drinking Water Standards. Where metal concentrations in the wells exceeded regulatory criteria, exceedences appeared in both up- and down-gradient wells suggesting background metal concentrations as opposed to significant leaching from the WFS. The WFS did not result in a negative environmental impact on the site. State environmental regulatory agencies were the most frequently identified source of barriers to the beneficial reuse of WFS and reuse is further compounded by the lack of decision-based scientific tools such as life-cycle or risk-based analysis methods. The resultant liability exposure from state and federal regulations was the prevailing concern expressed by Departments of Transportation (DOTs) when considering using a regulated waste such as WFS. Furthermore, foundries often lack an organizational commitment to the reuse of its WFS as evidenced by the lack of both product quality control and a marketing strategy. To facilitate the use of WFS by INDOT, an Acceptance Criteria protocol based on the Microtox™ bioassay test was developed as part of this project. Additionally, a proposed 'Recurring Special Provision' for the use of WFS in embankment construction and a WFS stockpile random sampling protocol were developed. The necessary decision tools for incorporating WFS into transportation construction, primarily embankment construction, appear to be available to the Indiana DOT (and other DOTs) as products of this research.			
17. Key Words Waste foundry sand; highway embankments; bioassay; Microtox™; environmental quality; regulatory issues; liability; cost savings		18. Distribution Statement No restrictions. This document is available to the public through the National Technical Information Service, Virginia, 22161	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 201	22. Price

TABLE OF CONTENTS

	Page
LIST OF TABLES.....	vi
LIST OF FIGURES	viii
IMPLEMENTATION SUGGESTIONS	xii
CHAPTER 1: INTRODUCTION.....	1
CHAPTER 2: OBJECTIVES.....	7
2.1 Overview.....	7
2.2 Specific Research Objectives.....	7
CHAPTER 3: TECHNICAL AND REGULATORY BACKGROUND.....	9
3.1 Permanent Pattern/Expendable Mold Casting	9
3.2 Regulatory Issues	16
3.2.1 Introduction.....	16
3.2.2 Federal Hazardous and Solid Waste Criteria.....	17
3.2.3 Indiana Solid Waste Regulations	20
3.2.4 Comparison of Various Foundry States' Solid Waste Regulations and Beneficial Reuse Criteria	22
3.2.5 Reuse Alternatives and State DOT Reuse Activities	29
3.2.5.1 <i>Pennsylvania</i>	30
3.2.5.2 <i>Michigan</i>	32
3.2.5.3 <i>Wisconsin</i>	32
3.2.5.4 <i>Ohio</i>	34
3.2.5.5 <i>Iowa</i>	35
3.2.5.6 <i>Illinois</i>	36
CHAPTER 4: WASTE FOUNDRY SAND LIABILITY ISSUES	37
4.1 Introduction.....	37
4.2 Potential INDOT Liability When Using Waste Foundry Sands.....	37
4.3 Liability Under CERCLA (Superfund).....	46
4.4 CERCLA (Superfund) Liability Reform	47

CHAPTER 5: ENVIRONMENTAL CHARACTERIZATION OF WASTE FOUNDRY SAND	50
5.1 Prior Chemical Characterization of Foundry Wastes	50
5.2 Bioassay Characterization of Waste Foundry Sand.....	57
5.2.1 Introduction.....	57
5.2.2 Microtox™ Bioassay	61
5.2.3 Microtox™ Bioassay Testing of Foundry Wastes.....	66
5.2.3.1 <i>Sensitivity of the Microtox™ Bioassay to Chemicals Typically Found in WFS' Leachates</i>	70
CHAPTER 6: CR 206 FIELD DEMONSTRATION PROJECT	73
6.1 Introduction.....	73
6.2 Geotechnical Work Plan	76
6.3 Environmental Work Plan.....	79
6.3.1 Methods and Materials.....	83
6.3.1.1 <i>Introduction</i>	83
6.3.1.2 <i>Bioassay Leachate Generation</i>	84
6.3.1.3 <i>Microtox™ Testing Protocol</i>	86
6.3.1.4 <i>Nitrotox Testing Protocol</i>	87
6.3.1.5 <i>Ion Chromatography Testing Protocol</i>	88
6.3.1.6 <i>Inductively Coupled Plasma Spectrophotometer Testing Protocol</i>	88
CHAPTER 7: RESULTS AND DISCUSSION.....	90
7.1 Geotechnical Results.....	90
7.2 Environmental Results	94
7.2.1 Introduction.....	94
7.2.2 Microtox™ Testing.....	94
7.2.3 Nitrotox Testing	100
7.2.4 Ion Chromatography Testing	103
7.2.5 Inductively Coupled Plasma Spectrophotometer Testing.....	110
7.3 Economic Considerations of Waste Foundry Sand Reuse.....	136
7.4 INDOT Acceptance Criteria for Waste Foundry Sand Reuse Based on the Microtox™ Bioassay Test	138
7.5 Random sampling of a Lot Within a Waste Foundry Sand Stockpile	140
CHAPTER 8: CONCLUSIONS AND RECOMMENDATIONS	143
LIST OF REFERENCES	147
APPENDICES.....	
Appendix A: Microtox™ 90% Comparison Test Protocol.....	153
Appendix B: Ion Chromatography and Operation of the GP40	159

Appendix C: Inductively Coupled Plasma Spectrophotometer Test Protocol.....	164
Appendix D: Random Sampling of a Lot Within a Waste Foundry Sand Stockpile, Indiana Department of Transportation, April, 1998	170
Appendix E: Acceptance Criteria for Waste Foundry Sand Reuse Based on the Microtox™ Bioassay Test, Indiana Department of Transportation, April, 1998	174
Appendix F: Proposed Recurring Special Provision for Embankments Constructed of Spent Foundry Sand, August, 1998	196

LIST OF TABLES

Table	Page
1: Summary of Selected Permanent Pattern/Expandable Mold Casting Processes	14
2: Federal Criteria Contaminants for Toxicity Characterization	19
3: Indiana Restricted Waste Criteria for Parameters Using the TCLP and the EP Water Tests.....	21
4: State Industrial Waste Classification Systems and Beneficial Reuse Testing Requirements	24
5: Lysimeter and Leachate Parameter Concentration Ranges from Ferrous Foundry Landfills.....	53
6: Organic Chemicals Measured in Waste Foundry Sands.....	55
7: Maximum Contaminant Concentrations for WFS Samples for Four Green Sand Molding Processes	58
8: Microtox™ Toxicity of Foundry Wastes.....	68
9: Microtox™ Toxicity of Foundry Wastes Upon Addition of EDTA.....	68
10: Contaminants Detected in WFS: Regulatory Levels, Lowest Reported EC ₅₀ Values, and Maximum Concentrations Detected in Leachate Analyses.....	72
11: Summarized Costs and Savings Data for the CR 206 WFS Embankment Project	139
12: Random Number Table.....	171
13: Random Number Table Example.....	173

Table	Page
14: Example Microtox™ Test Data (Acceptance of WFS Source)	183
15: Example Microtox™ Test Data (Rejection of WFS Source)	186
16: Example Microtox™ Test Data (Rejection of WFS Source, Non-Homogeneous Variances)	189
17: Virgin Sand Microtox™ Comparison Data	193

LIST OF FIGURES

Figure	Page
1: Permanent Pattern/Expendable Mold Process	11
2: Casting Process and Waste Stream Sources	15
3: Overview of Microtox™ Testing Protocols	62
4: CR 206 Project Section Layout	74
5: Geotechnical Instrumentation	78
6: Groundwater Monitoring Wells.....	80
7: Lysimeter Layout: Cross-Section	81
8: Field and Laboratory Compaction Testing of Auburn WFS	91
9: Horizontal Displacements of East Vertical Inclinometer	93
10: T5 Microtox™ Inhibition (Toxicity).....	95
11: T5 Microtox™ Inhibition (Toxicity).....	96
12: T15 Microtox™ Inhibition (Toxicity)	97
13: T15 Microtox™ Inhibition (Toxicity)	98
14: Nitrotox Inhibition (Toxicity).....	101
15: Nitrotox Inhibition (Toxicity).....	102
16: Ion Chromatography (IC) for Fluoride (F ⁻)	104
17: Ion Chromatography (IC) for Fluoride (F ⁻)	105

Figure	Page
18: Ion Chromatography (IC) for Chloride (Cl^-)	106
19: Ion Chromatography (IC) for Chloride (Cl^-)	107
20: Ion Chromatography (IC) for Nitrate (NO_3^- -N).....	108
21: Ion Chromatography (IC) for Nitrate (NO_3^- -N).....	109
22: Ion Chromatography (IC) for Sulfate ($\text{SO}_4^{=}$)	111
23: Ion Chromatography (IC) for Sulfate ($\text{SO}_4^{=}$)	112
24: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Silver (Ag).....	114
25: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Silver (Ag).....	115
26: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Barium (Ba).....	116
27: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Barium (Ba).....	117
28: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Nickel (Ni).....	118
29: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Nickel (Ni).....	119
30: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Arsenic (As)	120
31: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Arsenic (As)	121
32: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Chromium (Cr)	122
33: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Chromium (Cr).....	123

Figure	Page
34: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Copper (Cu).....	124
35: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Copper (Cu).....	125
36: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Lead (Pb).....	126
37: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Lead (Pb).....	127
38: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Selenium (Se).....	128
39: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Selenium (Se).....	129
40: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Zinc (Zn).....	130
41: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Zinc (Zn).....	131
42: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Cadmium (Cd).....	132
43: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Cadmium (Cd).....	133
44: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Manganese (Mn).....	134
45: Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Manganese (Mn).....	135
46: Microtox™ Response to Virgin Sands	141
47: Microtox™ 90% Comparison Test, Template 1	156
48: Microtox™ 90% Comparison Test, Template 2.....	157
49: Microtox™ 90% Comparison Test, Template 3.....	158

Figure	Page
50: Stratified Sampling of Sublots.....	172
51: Sublot Sample Location.....	172
52: Acceptance Criteria Elements.....	177

IMPLEMENTATION SUGGESTIONS

This research primarily focused on the geotechnical and environmental performance of a demonstration highway embankment constructed with WFS from a ferrous foundry. Results after two years of inservice evaluation indicate the embankment is performing satisfactorily and that the WFS are comparable to natural sand as fill material except the WFS are not considered freely draining. The WFS used in this project have not resulted in a negative environmental impact to the site. The following suggestions are offered to implement the results of this research:

- To facilitate the use of WFS by the Indiana DOT, an Acceptance Criteria protocol based on the Microtox™ bioassay was developed based on common virgin construction and foundry sands. It is suggested this protocol be used to screen WFS for future reuse applications. As additional information becomes available on the quality of WFS in Indiana, it is suggested acceptance criteria parameters be reevaluated and modified accordingly.
- Although the Microtox™ bioassay can measure the inhibition (toxicity) resulting from metal contamination, it is generally recognized as being more sensitive to organic contaminants. For WFS having borderline metal concentrations (i.e., approximating the upper-limit of the IDEM Type III criteria), additional metal specific testing should be considered to verify the metal concentrations of the WFS. It is suggested that samples collected for Microtox™ testing could also be used to verify metal concentrations using the appropriate regulatory leaching protocol. Although testing for metals is part of current regulatory testing for waste classification, regulatory testing requirements/results may not be representative of the WFS intended for reuse. For borderline WFS sources the user should consider indemnification from the

foundries. To ensure representative sampling of a WFS stockpile, a random sampling protocol was developed as part of this project. An attempt to address borderline Type III WFS has been included in the Proposed Recurring Special Provision in Appendix F.

- A proposed ‘Recurring Special Provision’ for use of acceptable WFS in embankment construction was developed, as part of this research effort, to allow the Indiana DOT to routinely permit the use of WFS as an alternate embankment material. This special provision should be submitted to the INDOT Specification Committee for acceptance and inclusion in contracts.
- Initial results for the prototype Nitrotox bioassay used in this research effort suggests it may be more sensitive to contaminants found in WFS than the established Microtox™ bioassay. Continuing research is addressing this premise. If the Nitrotox bioassay is found to be more sensitive it should be considered for use as a screening tool in lieu of or in addition to the current Microtox™ bioassay.
- Foundries should be encouraged to make their waste streams more uniform and acceptable to end users. Quality control measures should be considered. End uses should also address any liability or health/PPE issues resulting from worker (e.g., DOT and contractor personnel) exposure to WFS during construction. This information could assist foundries in monitoring and/or adjusting their own processes such that beneficial reuse of foundry residuals can be made more universally possible. For instance, foundries may be able to reduce the fine content of their WFS and resultant liability exposure by excluding baghouse fines as part of the WFS waste stream. Follow-up research is addressing some of these concerns.
- Field demonstration projects using foundry wastes for controlled low-strength material (‘CSLM,’ or flowable fill) aggregate are also recommended, particularly based on the INDOT-funded research effort completed by Bhat and Lovell (1996). Such projects

could be easily monitored, and the option of using waste foundry sand in flowable fill may be especially attractive to foundries which are unable to produce the large quantities of WFS required for highway projects.

The necessary decision tools for incorporating WFS into transportation construction, primarily embankment construction, appear to be available to the Indiana DOT (and other DOTs) as products of this research.

CHAPTER 1: INTRODUCTION

Foundries in the United States produce over 9 million Mg of ferrous castings per year (Clegg, 1991) and dispose of waste foundry sands (WFS) at a national average of approximately one Mg of sand wasted per Mg of casting produced (McIntyre, 1992). In Indiana alone, the Indiana Cast Metals Association (INCMA) estimate over 408,000 Mg of WFS are produced annually (INCMA, 1992).

WFS are a byproduct of the permanent pattern/expendable mold process, and are an enormous source of solid waste which must be discarded by the foundry industry. The majority of WFS are deposited in restricted (non-hazardous, solid waste) or sanitary waste landfills. A permanent pattern/expendable mold process is simply described as the use of expendable sand molds, shaped by reusable patterns and hardened through the use of compaction and added binders. Molten metal is poured into the resulting molds which determine the exterior shape, while interior cavities are obtained by placing sand cores inside the mold cavity prior to pouring the metal. As the binder coated sand is exposed to extreme temperatures during metal pours, its physical and chemical properties deteriorate with time (Salazar, 1997).

Fresh raw sands are typically brought into foundries on a daily basis as makeup sand for spent residuals and will pass through many cycles of internal reuse before being finally discarded. Many foundries practice sand recycling and sand reclamation (especially in Europe and Japan) as ways of reducing costs by reducing the volumes of both new sand purchased and spent sand disposed (Ziegler, 1994). Setting aside the fact that the feedstock metals brought into these plants are themselves largely waste materials (i.e.

engine blocks, used rail lines, etc.), internal recycling of core and molding sands is an important aspect of any plant's overall survival in an extremely competitive business (Bastian, 1996).

Recycling involves an evaluation of the WFS properties (e.g. grain size, grain shape), with a large portion of the sand determined to be suitable for reuse within the foundry. Sand reclamation involves mechanical and thermal processes to recondition the sand including screening for proper grain size distribution. In countries like Japan, where space limitations make it very difficult to dispose of wastes, foundries using sand reclamation have reported sand wasted per casting produced ratios of, typically, about 0.2 (McIntyre, 1992), a number lower than the ratio of 1.0 (spent sand: cast metal) reported as the national average (Salazar, 1997). While reclamation reduces the volume of waste streams leaving the foundries, residuals from reclamation processes can include concentrated amounts of binders and additives which may represent increased disposal costs due to waste reclassification (CISA, 1995). While WFS comprise the majority of a foundry's waste stream, other materials and processes within foundries including cleaning and grinding operations, slag, and dust collector equipment such as bag houses (Krueger, 1989) produce wastes which need to be disposed of and are often mixed with, and considered to be part of the WFS.

Historically, WFS were used by many cities surrounding large foundry operations as a common construction material for fills and foundations. As environmental regulations became more stringent and liability issues arose with regard to producing or reusing special wastes, the destination of these wastes changed from constructive reuses to sanitary landfills, foundry-owned monofills, and other waste disposal locations (Salazar, 1997).

In 1976, the Resource, Conservation, and Recovery Act (RCRA) was passed which defined a solid waste (e.g. WFS) as well as a hazardous waste. RCRA generally applies to the generation, transportation, treatment, storage and disposal of hazardous waste (42

U.S.C. § 9601). With passage of the 1980 Comprehensive, Environmental Response, Compensation, Recovery Act (CERCLA), better known as Superfund, and its subsequent amendments, the government and private parties, in some instances, were authorized to take action in response to the “release or substantial threat of release” of any hazardous substance regardless of the effect of the release on the environment, or of “any pollutant or contaminant which may present an imminent and substantial danger to the public health or welfare” (42 U.S.C. § 9604(a)(1)). Liability under Superfund became very broad in its scope of enforcement and in an effort to find deep pockets to pay for remediation costs, administrators and judges have had incentives to further broaden the scope of liability.

While foundries point to the apparent benefits, possibilities, and resultant savings from reusing WFS, as well as the historical precedents, current and future end users will frankly have to address the legal reality of environmental liability which any waste material reuse might impose. Indeed, State-level Department of Transportation (DOT) officials contacted in the Midwest (e.g. Wisconsin, Ohio, Iowa, Illinois, and Indiana) commonly characterized the issue of liability as a significant, if not the dominant hurdle, which has yet to be resolved to successfully reuse foundry sand. This concern of being connected (legally and financially) to the title of, ‘potentially responsible party’, is understandably worrisome and reflects the current conservative DOT mindset regarding the reuse of WFS. While these sands may truly be “*cleaner than dirt*,” as typically claimed by foundry representatives, potential end users are nonetheless compelled to develop their own ‘*reasonable engineering certainty*’ regarding long-term acceptability [and resultant liability] (Bastian, 1996).

Because of the spectre of liability, some WFS reusers have required indemnification from a foundry as a prerequisite to using the foundry’s WFS. Indemnification clauses, however, may represent an added cost and some argue a potential additional exposure for the foundry. For instance, permitted landfills may offer additional protection from a ‘contaminant release’ through the use of liners, leachate collection systems, etc., while a job site, such as a highway embankment, would not likely offer such safeguards.

Generally, there would be fewer controls and less protection at a job site than at a permitted landfill. While this realization underscores the importance of adequately characterizing the suitability of a waste for its intended reuse, a contaminate release from a job site, even though possibly not resulting from use of the WFS, could potentially become the responsibility of the foundry to remediate. Conversely, it may also be argued that foundries assume liability for any future landfill environmental problems while having no control over landfill management and the use of spent sand as a raw material in reuse applications greatly reduces that liability (CISA, 1995; Krueger, 1989).

As landfilling of WFS became increasingly unattractive, foundries sought assistance and relief on federal and state legislative levels. In 1991, the U.S. Congress passed the Intermodal Surface Transportation Efficiency Act (ISTEA) which directed the U.S. DOT and EPA to conduct studies on the reuse of recycled materials in highway construction. Also in 1991, the Indiana General Assembly required INDOT and Purdue University to perform a feasibility study on reusing six waste streams including: coal combustion byproducts, waste tires, ebonite materials (found in waste batteries), waste foundry sand, recycled asphalt pavement, and building demolition materials for use in road construction projects undertaken by the department.

Efforts are being made to reestablish WFS reuse activities. Extensive geotechnical laboratory investigations of the engineering properties of WFS have been performed. Research results of sands from gray and ductile iron (ferrous) foundries indicate the WFS are potentially suitable, from a geotechnical standpoint, for various uses in highway construction, such as embankments, subgrade, and flowable fill (Javed, 1993; Javed and Lovell, 1994; Javed, 1994; Mast, 1997). Other uses of WFS have been proposed as well, including daily landfill cover and aggregate applications in concrete, asphalt, bricks and tile (CISA, 1995).

A series of studies on the constituents found in foundry sand wastes and their concentrations suggest many WFS can also safely be reused from an environmental

perspective. Research (Bastian, 1996) funded by the Indiana Department of Transportation (INDOT) and INCMA demonstrated the capability of a complementary (to regulatory testing) Microtox™ bioassay (90% Comparison Test) to screen the ‘toxicity’ of WFS. The Microtox™ bioassay is an existing, commercially available bioassay developed and marketed by Microbics Inc. The Microtox™ bioassay uses surrogate biological indicators (*Vibrio fischeri* bacteria) and can be used to effectively ‘fingerprint’ and measure the ‘toxicity’ of WFS, as compared to natural (virgin) construction and foundry sands. Microtox™ bioassay testing indicated the majority of WFS from ferrous foundries were less inhibiting (‘toxic’) than natural sands, in part supporting industry claims their wastes are ‘cleaner than dirt’. These WFS satisfy INDOT reuse acceptance criteria (INDOT, 1998) as well as the Indiana Department of Environmental Management (IDEM) beneficial reuse criteria (Indiana Administrative Code (IAC), 329 IAC 10-9-4 for a Type III and Type IV waste).

WFS reuse can be beneficial to all parties involved. Foundries gain significant economic savings by extending the service life of existing foundry owned landfills and by avoiding or delaying stringent permitting requirements and costly permitting fees for siting new landfills. Additionally, generators are often willing to provide WFS to a job site at no cost to the end user in lieu of incurring landfill tipping fees. Consequently, end users, such as contractors and DOTs, can obtain a free or very low-cost source of sand (fine aggregate). Little capital investment typically is required by the foundries to make their WFS acceptable for reuse applications. Transportation costs for hauling WFS, however, generally dictate the need for a relatively close proximity between the foundry and the job site. Where landfill tipping fees are higher, as in the northeastern states, foundries are willing to transport their WFS longer distances. Local governments, trash haulers, and local citizenry also benefit from the constructive reuse of WFS as WFS are diverted from sanitary landfills thereby extending landfill service lives, and avoiding potential increases in hauling costs and transfer fees as the landfills reach capacity and new sites must be found.

With laboratory evaluations complete, indicating that WFS from ferrous foundries could provide the necessary engineering properties for a highway embankment and that the Microtox™ test could screen the toxicity of the WFS to prevent a negative environmental impact on the site, the next step was the construction of a full scale WFS embankment, with geotechnical and environmental instrumentation installed to evaluate performance. In July and August of 1996, 42,815m³ of WFS from the Auburn Foundry Inc. monofill (single waste landfill) in Auburn, Indiana was placed as structural fill in a 105 m long embankment section on County Route (CR) 206 in DeKalb County, Indiana under INDOT Contract R-22343-A. The Auburn monofill is located 27 km from the project site and is not covered, allowing 'weathering' of the sand.

The Auburn Foundry is a large gray iron foundry which uses a phenolic urethane compound as the principal core binder. The phenolic urethane binder belongs to the 'cold box organic binder' group and includes a combination of phenol formaldehyde resin and polymeric isocyanate. The catalyst is vaporized before application to produce a rapid cure, and typical catalysts are gaseous TEA (triethylamine) or DMEA (dimethyl-ethylamine) (Bastian, 1996; Clegg, 1991). The daily metric tons of metal cast (≈ 907 Mg) and spent sand landfilled indicate the Auburn plant was operating with a WFS to cast metal ratio (by weight) of 0.125-0.15. While the efficiency of recycling for the plant is almost an order of magnitude greater than the U.S. national average, the daily disposal was still greater than one hundred metric tons per day, demonstrating the need for constructive reuse alternatives (Salazar, 1997).

CHAPTER 2: OBJECTIVES

2.1 Overview

The overall objective of this project was to assist INDOT in the planning, construction, monitoring, and evaluation of a prototype highway embankment built using WFS from a ferrous foundry which satisfied the INDOT and IDEM solid waste and beneficial reuse criteria. This report primarily addresses the environmental performance and to a lesser degree the geotechnical performance of the fill materials and the project site through two years of in-service performance. For a detailed discussion of the geotechnical evaluation and performance, the reader is directed to the companion report for this project (Fox and Mast, 1998). Regulatory and liability issues and, to a lesser degree, quality control and economic issues surrounding WFS reuse applications are also discussed in this report.

2.2 Specific Research Objectives

- To perform Microtox™ bioassays on leachates from the stockpile and job site WFS samples to determine initial inhibition levels and to evaluate the relative homogeneity and 'toxicity' of the WFS over time. To compare these results with established acceptance criteria to determine the suitability of the WFS for beneficial reuse applications.
- To perform Microtox™ and Nitrotox bioassays and selective ion and metal testing on samples from six groundwater wells around the site, over an extended period of time, to determine background inhibition levels and concentrations and the resulting environmental impact on the site from the use of the WFS.

- To perform Microtox™ and Nitrotox bioassays and selective ion and metal testing on samples from two lysimeters, one placed in the WFS embankment and one in the natural (virgin) sand embankment, over an extended period of time to determine differences in on-site leachate characteristics and the resulting impact on down-gradient wells.
- To investigate the migration of potential contaminants from the stockpile and job site WFS into lysimeter leachates and groundwater samples.
- To investigate the correlation between results from laboratory leachates, and field lysimeter/well samples in an effort to further assess the accuracy of laboratory methods in estimating complex field leaching conditions.
- To investigate the correlation between the prototype Nitrotox bioassay and the established Microtox™ bioassay in their ability to characterize the ‘toxicity’ of the WFS.
- To evaluate the geotechnical performance of the WFS as a suitable embankment material.
- To examine regulatory and liability issues affecting WFS reuse applications by Departments of Transportation.

CHAPTER 3: TECHNICAL AND REGULATORY BACKGROUND

3.1 Permanent Pattern/Expendable Mold Casting

Foundry sand is utilized in permanent pattern/expendable mold casting processes. In these processes, molten metal is poured into a mold made of sand which has been shaped and hardened to withstand the pressure and heat derived from the heated metal. After the metal has cooled, the sand is separated from the casting and recycled. Although foundries go to great lengths to recycle as much sand as possible, some foundry sand must be wasted each cycle due to the physical and chemical breakdown of the sand and from the necessity of using virgin sand for some parts of the mold (Clegg, 1991). Some factors which determine the amount of used sand that must be replaced by virgin sand include: amount of burned binder on the sand, which reduces re-bonding capabilities; presence of unburned core binder; presence of relatively strong screened-out agglomerates; and deteriorated grain size/shape, indicating it is no longer suitable to ensure a good casting surface finish (McIntyre, 1992; FM&T, 1996b). The Casting Industry Suppliers Association (CISA) sand committee task force defines spent foundry sand as “sand that has given up all its useful value to the foundry process, ...sand that has already been recycled countless times and eventually has to be removed since it no longer meets specifications for making quality castings” (CISA, 1995).

Several types of virgin sands are used in permanent pattern/expendable mold casting and can be classified into four groups: silica, olivine, chromite, and zircon. The most commonly used sand is silica sand. Each group has several subgroups according to the particular characteristics of the sand. Properties of concern for a foundry to determine the best sand to use are dictated by process needs and include: grain fineness and distribution, grain shape, chemical purity especially in regards to silica content, loss of

ignition (LOI), density, acid demand value (ADV), pH, clay and moisture content, sand temperature characteristics, permeability, and other considerations (Ziegler, 1994).

Clays are added to the mold sand for casting. When the clays, which are hydrous aluminosilicates such as illites, kaolinites, and montmorillonites, are hydrated, surface interactions between sand and clay particles produce the bonding forces. Other additives, such as seal coal, starch, pitch, asphalt, and petroleum distillates may be used to control strength, deformation characteristics, surface finish, and reduce defects (Clegg, 1991). Sea coal is a very highly volatile bituminous coal which is finely ground and then mixed and mulled with the molding sands to help prevent defects in the metal casting. It is typically used in sand systems for gray iron, ductile iron, and malleable iron castings (Javed, 1994).

When casting metal in a pattern/expendable mold casting process, the first pieces created are the patterns (Figure 1). These pieces are fashioned in the shape of the desired casting, and can be made from wood, plastic, or metal. The pattern is usually made in two parts, corresponding to the top and bottom parts of the mold. The next step is the fabrication of the two parts of the mold. Box shaped containers are filled with sand, the pattern pieces are placed against the sand surface, and the sand is compacted by impact, squeezing, vibration, airflow, or vacuum. The pattern is then removed, and the two parts of the mold can be fastened together to create the cavity into which the metal is poured (Clegg, 1991).

Most permanent pattern/expendable mold casting processes use 'green sand' meaning that the sand/water mixture is used while still damp, and is usually made up of a combination of recycled/reclaimed sand and virgin sand (Clegg, 1991). The major components of 'green sand' are typically mixed in the following proportions (Javed, 1994):

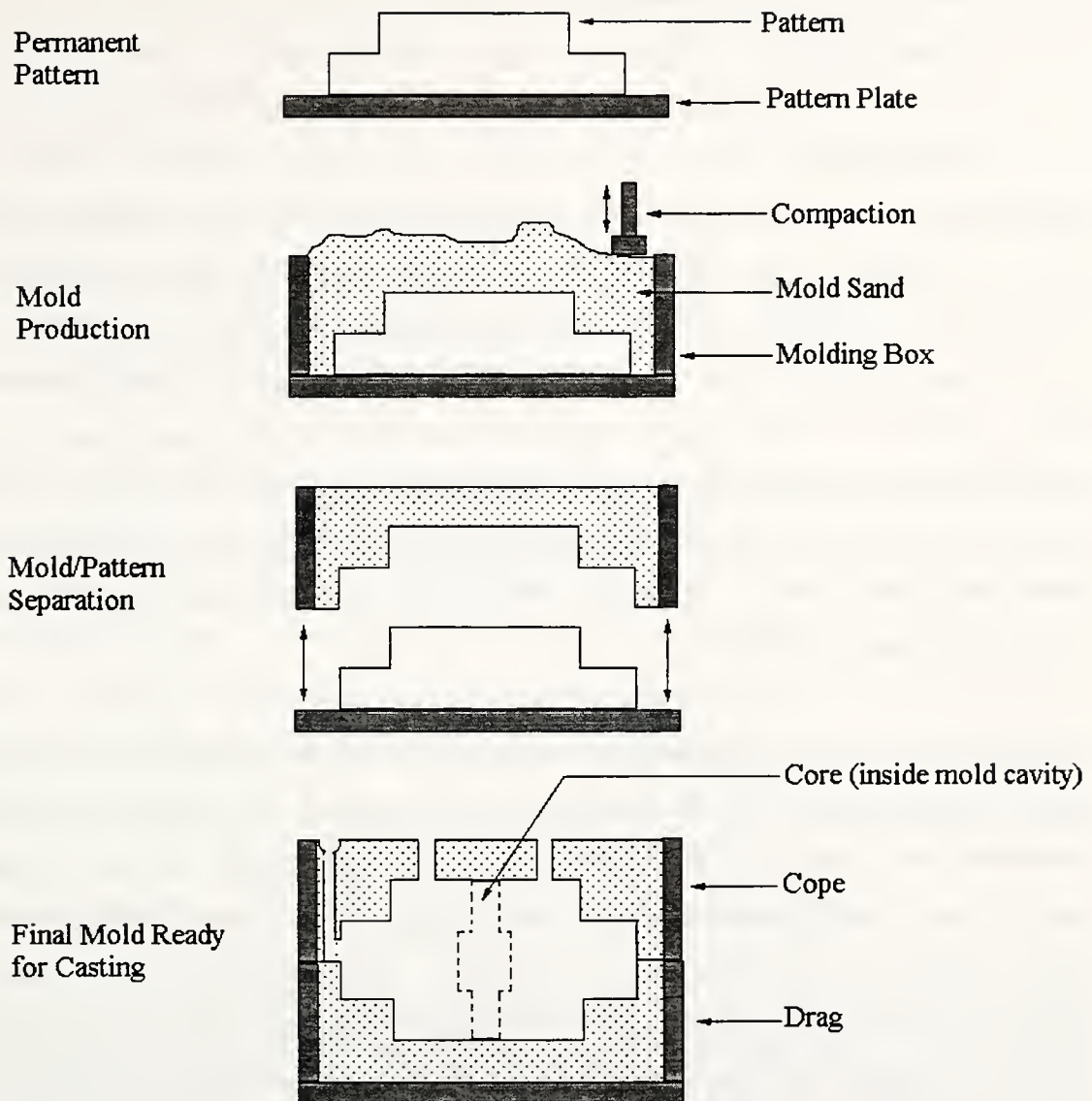


**Figure 1 – Permanent Pattern/Expendable Mold Process
(Redrawn from Clegg, 1991)**

- 85 to 95% uniform quartz sand,
- 4 to 10% bentonite clay,
- 2 to 10% combustible additives (e.g. sea coal), and
- 2 to 5% water.

When the metal is poured and the sand reaches temperatures above 100°C, free moisture is driven off. Many organics, such as those found in sea coal, are then volatilized (FM&T, 1993). Above approximately 600°C, the combined water which forms the sand-clay and clay-clay bonds begins to be driven off; all water is gone at temperatures above 800°C. After the casting has cooled, the sand can be separated (usually by shaking/vibratory mechanisms), and recycled: fine particles are removed, lumps of sand are broken up, any remaining metal is removed, and the sand returned to the stockpile of system sand (FM&T, 1993). As much as 90 to 95 percent of the sand is recycled after each casting (Bastian, 1996).

Permanent interior cavities are formed by placing sand cores at appropriate locations inside the mold cavity prior to pouring the molten metal. Core pieces are made exclusively from virgin sand. This is necessary as the chemicals used as binders for the cores are not effective with clay-coated sand (Clegg, 1991). Some typical binders include vegetable and petroleum oils, sodium silicate, ground corn flour and oil, ground hardwood cellulose, and synthetic binders like phenol formaldehyde, phenol isocyanate, and alkyd isocyanate. The amount of binder used in the sand is determined according to casting temperatures and binder performance: too much binder causes clumps, interfering with sand reclamation and allowing excess amounts of contaminants to remain in the spent sand; not enough binder, on the other hand, could result in lack of the strength needed to undergo the casting process and perform properly (Krueger, 1989).

Although there are many varieties of chemical binder systems, the majority of binders used in Indiana ferrous foundries are mixtures of organic chemicals. Organic binders can be grouped into three categories: cold set (also called no-bake), cold box, and hot box. Most of these binders utilize one or more binder chemicals and a catalyst or hardener

which, when added, promotes the binding reaction(s). Cold set binder systems utilize liquid catalysts, and reactions occur at room temperature. Cold box binder systems utilize gaseous catalysts, again at room temperature. Hot box binder reactions take place at high temperatures (150 to 300°C) (Clegg, 1991). A short summary of the different binder types is given in Table 1. Silicate – CO₂ binders, alumina phosphate binders, and furan/furfuryl alcohol based binders, in particular, have been promoted as being environmentally friendly due to the lack of toxic volatiles created by core making and casting processes (Bambauer, 1993; FM&T, 1993).

While broken cores and discarded molding sands are collectively called WFS or spent foundry sand, foundry solid wastes typically include refractories, annealing and cleaning room wastes, slag, coke ash, scrubber discharge, baghouse dust, and floor sweepings. By volume, the majority of foundry wastes are comprised of WFS (Boyle, 1979). Waste stream sources are shown in Figure 2. It has been estimated that of all wastes disposed of by a foundry, between 65% and 99% originate as mold and core sands. Of these wastes, the portion disposed of as spent or waste foundry sands (WFS) is a combination of system sands (65-99%), core sand and core butts (2-19%), slag (0-16%), cleaning and grinding (0.5-9%), and dust collector wastes (0-11%) (Krueger, 1989).

One way to promote WFS reuse, as well as to reduce future liability, is by segregating waste streams as part of process quality control for foundry wastes. For example, baghouse dust generally contains relatively high concentrations of metals as compared to any other waste stream (Krueger, 1989). Segregating this waste stream out of the total waste can be beneficial in that while the baghouse dust may contribute only a very small percentage of the total waste volume, its segregation could eliminate the main source of a contaminant from the total waste. A plant in Michigan, for instance, which collects and treats WFS for constructive reuse, segregates wastes based on the metal cast at the point of origin, sends slag to a different facility for alternative treatment, and disposes only the pollution control streams with high metal concentrations and non-reusable fines (Walborn, 1997).

Table 1: Summary of Selected Permanent Pattern/Expandable Mold Casting Processes (Bastian, 1996)

Type	Name	Binders and Additives
Inorganic Option	Greensand	Clays, water, starch, and sea coal
	Alumina phosphate	Aluminum phosphate resin, and metal oxide hardeners
Cold Set/No-Bake Options	Furan	Furfuryl alcohol resins, urea, phenol, and aryl sulfonic acids
	Phenolic urethane	Phenol formaldehyde resin, isocyanates, and liquid amines
	Sodium silicate	Liquid sodium silicate and liquid organic ester
Cold Box Options	Phenolic urethane	Phenol formaldehyde resin, polymeric isocyanate, and gaseous amine
	Silicate-CO ₂	Liquid sodium silicate, coal dust, clays, and CO ₂ gas
Heat Activated Options	Hot Box	Furfuryl alcohol or phenolic resin, urea, formaldehyde, and acid catalyst
	Shell Molding	Phenol formaldehyde resins, calcium stearate, Vinsol, iron oxide, and hexamethylene tetra-amine
	Air Set	Various oil resins
	Core oil	Unsaturated oil resins, oxygen sources, and solvents

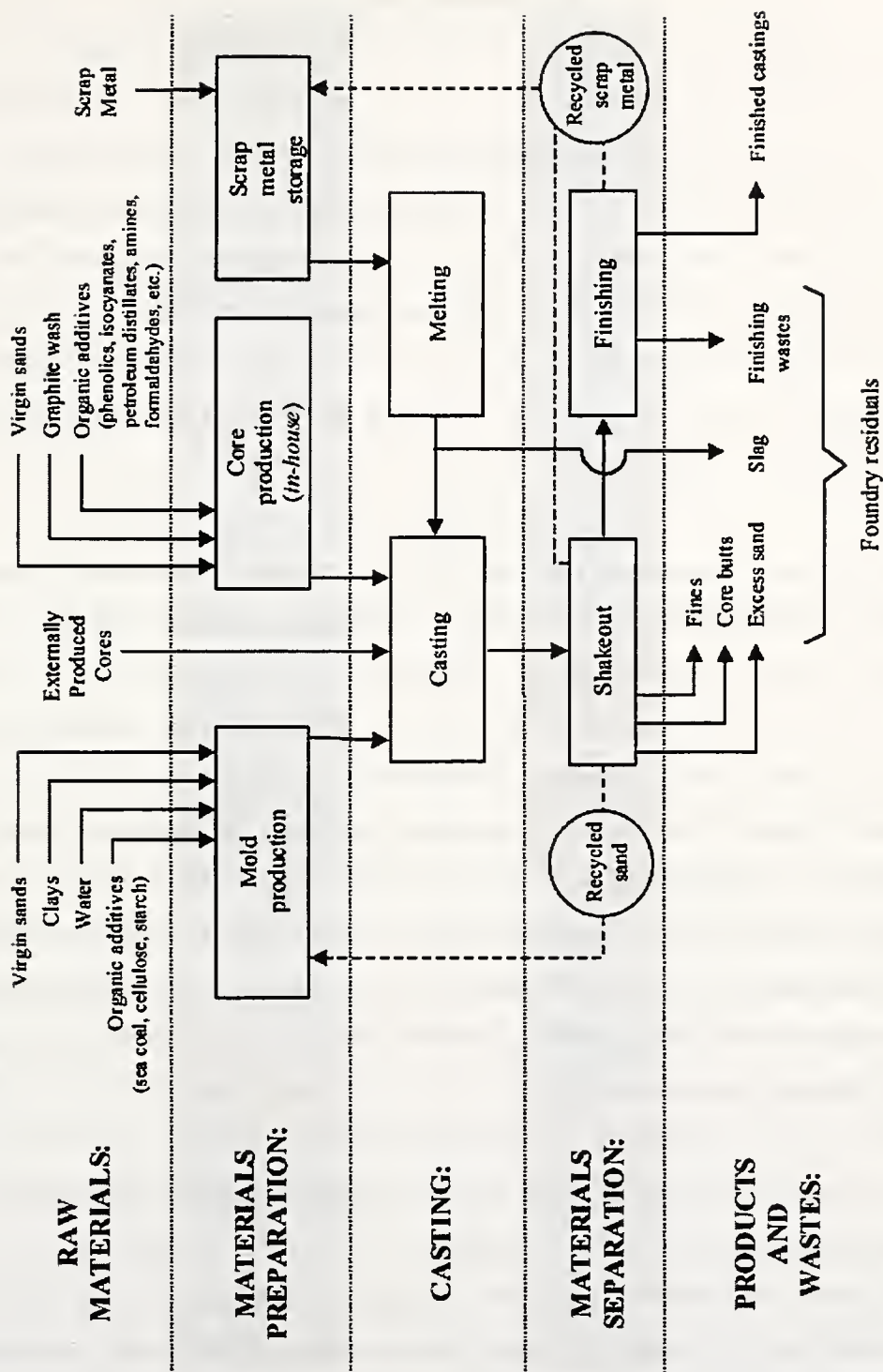


Figure 2 – Casting Process and Waste Stream Sources
(Bastian, 1996)

3.2 Regulatory Issues

3.2.1 Introduction

In spite of significant efforts by the foundry industry to develop options for the beneficial reuse of WFS, the actual amount of reused WFS lags behind the expectations of casting industry analysts and leaders. The result is a continuing drain on industry profits due to significant waste handling and disposal costs, including the societal cost of using limited landfill space to dispose of a product for which documented uses exist (Kauffmann, 1996).

Kauffman suggests there are both technical (e.g. engineering properties) and institutional barriers confronting the beneficial use of WFS. While the technical feasibility of reusing WFS from ferrous foundries has been supported by laboratory and field evaluations (Javed, 1993; Javed and Lovell, 1994; Javed, 1994; Mast, 1997), institutional barriers remain. Kauffman broadly defines institutional barriers as the result of non-technical issues such as market, business, and regulatory realities and perceptions, stating state environmental regulatory agencies are the most frequently identified source of institutional barriers to the beneficial reuse of WFS. Foundries believe regulatory agencies must balance the real and perceived environmental risks to groundwater and human health with the environmental and economic impacts of loaded landfills and cost burdened factories (Kauffmann, 1996).

While state regulatory agencies share the same goals of protecting groundwater quality and human health, there are numerous differences in how they classify WFS (e.g. solid or industrial waste, restricted wastes, special wastes, or a multi-level classification system), as well as differences in regulatory test procedures and standards, beneficial reuse criteria and permitting requirements. These differences will be discussed in more detail later.

3.2.2 Federal Hazardous and Solid Waste Criteria

With the passage of the Resource Conservation and Recovery Act (RCRA) of 1976, a standard approach to classify certain industrial or solid wastes, such as WFS, is based on a waste's characteristics. Under these federal rules, to be a hazardous waste, a substance must first be a 'solid waste' (42 U.S.C. 8 6903 (5)). RCRA defines a 'solid waste' as any 'discarded material' which is either:

- a) abandoned (by being disposed, burned, incinerated, or stored prior thereto),
or
- b) inherently waste-like, or
- c) a secondary material (i.e. any spent material, sludge, byproduct, etc.), when recycled in any of the following four ways:
 - 1) used in any manner constituting disposal:
 - 2) burned for energy recovery used to produce a fuel or contained in fuel;
 - 3) reclaimed; or
 - 4) accumulated speculatively.

A solid waste under RCRA is considered to be a hazardous waste if either listed by the Environmental Protection Agency (EPA) or exhibiting one of the four characteristics of ignitability, corrosivity, reactivity, or toxicity as defined in 40 CFR 261. The principal characteristic of concern with WFS is toxicity around which state regulatory classification criteria center to prevent leaching of potential toxic constituents into local groundwater. Toxicity is, typically, determined using the Toxicity Characteristic Leaching Procedure (TCLP) test, which generally replaced the Extraction Procedure Toxicity test (EP Tox) initially required by the EPA in 1980.

The original EP Tox test used a mixture of solid (e.g. WFS) to liquid (e.g. deionized water) ratio of 1:16, with the addition of 0.5N acetic acid as needed to maintain the pH of the solution close to 5.0 throughout the procedure. The protocol required an extract analysis of 8 metals and 6 herbicides (Salazar, 1997). The TCLP test is a similar test to the EP Tox test in terms of the leaching protocol, but the list of regulated chemicals

includes 25 organics, 8 metals and 6 insecticides/herbicides and compares the results with pre-established levels over which toxicity is triggered (Case, 1995).

It has been shown the TCLP test is more aggressive than the EP Tox test, yielding higher concentrations than those obtained using the EP Tox test. Additionally, the TCLP protocol includes a zero headspace extractor to avoid loss of volatile components during extraction, and the predetermination of the appropriate extraction fluid so that no pH adjustments are necessary during extraction (Javed, 1993). Table 2 is a listing of contaminants tested for using the TCLP protocol, along with respective maximum concentration values, (40 CFR 261). The maximum concentration levels for the 8 metals characterizing a waste as hazardous were traditionally set at levels of 100 times the National Primary Drinking Water Standards (NPDWS), but some variation on this practice has occurred as regulatory agencies have further evaluated individual chemicals and their resultant risks.

Two other leaching methods similar to the TCLP and EP Tox tests are used to characterize WFS toxicity, with the most significant difference being the lack of pH control during extraction. The EP Water test, also referred to as the Indiana leach test, follows the same protocol as the EP Tox test, but uses deionized water with no acetic acid addition and thus no pH control. The other leaching method used to characterize WFS is the American Foundrymen's Society (AFS) test. This procedure is also conducted using deionized water with no pH control, but attempts to simulate field conditions by exposing the waste to a continually fresh leaching medium by using a total of three elutions (Case, 1995; Javed, 1993).

The EP Tox and TCLP leaching tests were designed to reproduce the worst case scenario for environmental conditions present at a disposal site, such as a landfill containing municipal wastes, where acidic conditions can exist due to the interaction between different wastes. The EP water and AFS test, however, were developed to better replicate

**Table 2: Federal Criteria Contaminants for Toxicity Characterization
(40 CFR 261)**

Contaminant	Regulatory Level (mg/L)	Contaminant (continued)	Regulatory Level (mg/L)
Chlorinated Organics		Other Organics	
Carbon Tetrachloride	0.5	Benzene	0.5
Chlorobenzene	100.0	o-Cresol	² 200.0
Chloroform	6.0	m-Cresol	² 200.0
2,4-D	10.0	p-Cresol	² 200.0
1,4-Dichlorobenzene	7.5	Cresol	² 200.0
1,2-Dichloroethane	0.5	2,4-Dinitrotoluene	¹ 0.13
1,1-Dichloroethylene	0.7	Lindane	0.4
Hexachlorobenzene	¹ 0.13	Methyl ethyl ketone	200.0
Hexachlorobutadiene	0.5	Nitrobenzene	2.0
Hexachloroethane	3.0	Pyridine	¹ 5.0
Pentachlorophenol	100.0	Metals	
Tetrachloroethylene	0.7	Arsenic	5.0
Trichloroethylene	0.5	Barium	100.0
2,4,5-Trichlorophenol	400.0	Cadmium	1.0
2,4,6-Trichlorophenol	2.0	Chromium	5.0
Vinyl chloride	0.2	Lead	5.0
Agricultural Organics		Mercury	0.2
Chlordane	0.03	Selenium	1.0
Endrin	0.02	Silver	5.0
Heptachlor (and its epoxide)	0.008		
Methoxychlor	10.0		
Toxaphene	0.5		
2,4,5-TP (Silvex)	1.0		

¹Quantitation limit is greater than the calculated regulatory level. The quantitative limit therefore becomes the regulatory level.

²If o-, m-, and p-Cresol concentrations cannot be differentiated, the total cresol concentration is used. The regulatory level of total cresol is 200 mg/l.

the environment which these sands will be subjected to in a monofill or alternative-reuse site, where their exclusive presence will determine the leaching characteristics, as contaminants are extracted by percolating water (Bastian, 1996; Javed, 1993).

3.2.3 Indiana Solid Waste Regulations

In Indiana, WFS are regulated under Section 329 of the Indiana Administrative Code (IAC), Article 10, entitled “Solid Waste Land Disposal Facility Classification.” Part 4 of Rule 9 within this code (i.e., 329 IAC 10-9-4) specifies criteria used to characterize non-hazardous industrial solid wastes, also termed restricted wastes. Characterization criteria for WFS are based upon the TCLP test (329 IAC 3.1-6-1) and the Indiana Leaching Method (329 IAC 10-7-4), often referred to as the EP Water Test. Test criteria parameters are listed in Table 3.

In general, restricted wastes, such as WFS, must be disposed of in what are termed “Restricted Waste Sites.” These sites must conform to specifications more stringent than sanitary landfills but not as stringent as those for hazardous waste landfills (329 IAC 10-9-1). Certain exceptions apply, however, to WFS. WFS meeting IDEM Type IV criteria are excluded from regulation under 329 IAC 10-3-4. In addition, WFS meeting Type III standards are excluded from regulation under 329 IAC 10 when legitimately used, including as a base for road building (329 IAC 10-3-1 (14)). The vast majority of WFS from ferrous foundries meet Type III or IV criteria, and would be acceptable for use as road base construction materials according to Indiana regulations (Bastian, 1996). It should be noted resampling and testing of a foundry’s waste for classification must be completed every two years, whenever the process producing the waste changes, or according to a schedule determined by previous sampling and variability of the waste. A comprehensive list of organic additives used in the process producing the waste must be submitted with each test results.

While 329 IAC 10-3-1 (14) states that the “legitimate use of foundry sand which has been demonstrated as suitable for restricted waste site Type III under the provisions of 329 IAC 10-9-4, including the use as a base for road building” is excluded from the provisions of Indiana’s Solid Waste Rule, there was no clear definition of ‘roadbase’. In 1996, the Indiana Department of Transportation (INDOT) proposed the following definition for a

Table 3: Indiana Restricted Waste Criteria for Parameters Using the TCLP and the EP Water Tests^(a)

<u>TCLP (acidic leachate - mg/L)</u>				
Constituent	Type IV	Type III	Type II	Type I
Arsenic	≤0.05	≤0.5	≤1.25	<5.0
Barium	≤1	≤10	≤25	<100
Cadmium	≤0.01	≤0.1	≤0.25	<1.0
Chromium	≤0.05	≤0.5	≤1.25	<5.0
Lead	≤0.05	≤0.5	≤1.25	<5.0
Mercury	≤0.002	≤0.02	≤0.05	<0.2
Selenium	≤0.01	≤0.1	≤0.25	<1.0
Silver	≤0.05	≤0.5	≤1.25	<5.0

<u>EP Water Test (neutral leachate – mg/L)</u>				
Constituent	Type IV	Type III	Type II	Type I
Barium	≤1	≤10	≤25	*
Boron ^(b)	≤2	≤20	≤50	*
Chlorides	≤250	≤2,500	≤6,250	*
Copper	≤0.25	≤2.5	≤6.25	*
Cyanide, total	≤0.2	≤2	≤5	*
Fluoride	≤1.4	≤14	≤35	*
Iron	≤1.5	≤15	*	*
Manganese	≤0.05	≤0.5	*	*
Nickel	≤0.2	≤2	≤5	*
Phenols	≤0.3	≤3	≤7.5	*
Sodium	≤250	≤2,500	≤6,250	*
Sulfate	≤250	≤2,500	≤6,250	*
Sulfide, total	≤1	≤5	≤12.5	*
Total dissolved solids	≤500	≤5,000	≤12,500	*
Zinc	≤2.5	≤25	≤62.5	*
pH ^(c)	6-9	5-10	4-11	*

*Testing not required

^(a) Indiana Administrative Code (IAC), Section 329, Article 10, “Solid Waste Land Disposal Facility Classification,” Rule 9, Part 4 (i.e. 329 IAC 10-9-4)

^(b) Not included in 1996 Indiana Department of Environmental Management Edition

^(c) Acceptable Range (Standard Units)

roadbase to IDEM: “For the purpose of using foundry sands in highway applications roadbase shall be considered as any area lying within typical roadway boundaries where soil or aggregate is used subject to contract provisions and environmental siting criteria. This includes backfill materials over pipe, paving and embankment material, base, subbase, subgrade and special subgrade treatment, in essence, everything from the top of the pavement on down. Drainage layers and surface materials not encapsulated by asphalt or cement would normally be excluded.” IDEM indicated their concurrence with the roadbase definition on June 16, 1996.

As part of House Bill 1541 (1997), a new section was recently added to the Indiana Code (IC 13-19-3-7) which generally allows for the reuse of foundry sand that meets Type III classification, without the need for a permit. Effective July 1, 1997, the bill listed a series of applications for WFS, including but not limited to: daily landfill cover; protective cover for a landfill leachate system; capped embankments for landfill final cover, ground and sight barriers, or embankments for airports, bridges and overpasses, various structural fills; and as a raw material of several products including flowable fill, concrete, asphalt, brick, block, Portland cement, and others. A House Motion passed as part of the bill created a task force including representatives from special waste generators (including foundries) and representatives knowledgeable in the proposed reuse applications, to make recommendations to the Solid Waste Management Board on rules for reuse. This report by the task force is to be submitted to the Environmental Quality Service Council for use in making recommendations concerning the uses mentioned in IC 13-19-3-7 (Salazar, 1997).

3.2.4 Comparison of Various Foundry States’ Solid Waste Regulations and Beneficial Reuse Criteria

State regulatory agencies, in an effort to protect against potential environmental risks to groundwater and human health from industrial solid wastes, such as WFS, may adopt federal RCRA criteria or develop their own criteria as long as the resulting regulations are

as stringent as federal RCRA regulations. A comparison, however, of environmental regulations impacting industrial residual classification, test procedures and standards, beneficial reuse practices, and landfilling of WFS among major foundry states indicates numerous differences as well as similarities. For instance, a survey of 14 major foundry states' environmental regulatory criteria (Table 4) (Kauffmann, 1996) indicates a cross-section of these nuances.

Comparing how these states classify industrial wastes, eleven states start with the base classification of nonhazardous industrial waste as a solid waste or an industrial waste. For three states, Georgia, Iowa, and Tennessee, this is the only classification employed. Expanding upon the solid waste classification five states add a "special waste" classification within the nonhazardous waste designation. Six states use multi-level classification systems (a multi-level system involves a number of designated classes based on test parameters). Only Indiana uses a system which includes solid, special and multi-tiered classifications together. Two states, Ohio and Pennsylvania, employ the "residual waste" classification for some nonhazardous wastes and for Pennsylvania this is the only classification used. Finally, one state, California, has a more rigorous state hazardous waste definition and classification separate from the federal standard.

Generally, a waste generated by a manufacturing or industrial process, which is not hazardous is defined as an industrial solid waste. 'Special Waste' definitions, however, vary. Alabama defines a special waste as those wastes requiring special handling or disposal (e.g. WFS) as determined by the Alabama DEM. California classifies industrial

Table 4: State Industrial Waste Classification Systems and Beneficial Reuse Testing Requirements
(adapted from Kauffman, 1996)

Industrial Waste Classification Systems						Test Procedures and Standards for Beneficial Reuse Applicability					
	Solid Waste Category	Special Waste Category	Residual Waste Category	Multiple Tiers Within Category	State Hazardous Class.	TCLP or Acid Based Test	ASTM D3987 or Neutral Based Test	Use of Individual State Test System	Identification of Specific Test Levels	Additional Chemical Testing Beyond TCLP or Neutral Leachate Test	Bulk Analysis
Alabama	X	X				X			X		
California		X			X	X (c)		X		X	X
Georgia	X					X (d)					X
Illinois		X		X			X		X	X	
Indiana	X	X		X		X	X (b)		X	X	
Iowa	X					X			X		
Massachusetts	X	X				X					X
Michigan	X			X		X	X (a)		X	X	
New York	X					X					X
Ohio	X		X	X		X	X (a)		X		
Pennsylvania			X			X			X	X	X
Tennessee	X					X	X (a)		X		
Texas (e)	X			X		X	X		X	X	X
Wisconsin	X			X		X	X		X	X	X

(a) = Allowed if more representative of beneficial use.

(b) = Water leach test only for certain elements.

(c) = Used for RCRA hazardous determination only.

(d) = Bulk analysis required first and TCLP required based on bulk results.

(e) = Texas does provide for beneficial use without testing based on the material and the intended use.

special wastes based primarily on three tests: the TCLP, the California Waste Extraction Test (WET), and the Total Threshold Limit Concentration (TTLC) which is a total leach test.

WET evaluates organic and inorganic toxic substances based on a Soluble Threshold Limit Concentration (STLC). This results in a broader definition of a hazardous waste than the TCLP alone. The WET test was developed and implemented in California prior to EPA's development of the TCLP. A waste is federal hazardous based on RCRA and California hazardous based on STLC and TTLC test results. In general a material is eligible to be classified as a special waste if it tests below the STLC. Fourteen wastes are specified as eligible to be special wastes including foundry sand (Kauffman, 1996).

In Illinois, an industrial waste is classified as a special waste which is broadly categorized based on how it is produced rather than its specific content. Special wastes have many of the permit and legal requirements of hazardous wastes. For example, in Illinois, POTW sludge, paint sludge and bag house dusts are classified as special wastes and have to be disposed in an approved landfill with an approved permit. Massachusetts defines a special waste as having particular characteristics which require special controls in its handling and disposal to prevent an environmental impact. Examples include medical and asbestos wastes which are not eligible for beneficial reuse (Kauffman, 1996). In Indiana, a special waste is a non-hazardous waste based upon EP Tox test (or TCLP test) and neutral leachate test (EP water test) results.

As noted in Table 4, most of the surveyed foundry states use the TCLP test as the primary test for classifying a waste as well as whether the waste may be beneficially reused. In Alabama and Iowa this is the only test required. Illinois uses the ASTM D 3987 water leach test as the primary test while California uses both the TCLP and WET tests. Furthermore, seven states require additional chemical specific testing beyond the TCLP or neutral leachate tests and seven states require a total metals or bulk analysis in addition to leachate tests.

How the surveyed states determine beneficial reuse criteria, based on the testing results, also varies. For instance, in Alabama if a waste material is 50% of the TCLP standard, beneficial reuse is allowed if meeting beneficial use restrictions, while in Iowa WFS with leachate levels up to 90% of TCLP standards are considered acceptable for reuse. The WFS in Iowa must also have a pH between 5.0 and 10.0. In Pennsylvania, beneficial uses involving direct land contact are considered if test levels are less than or equal to 25 times groundwater standards (Kauffman, 1996).

In California, beneficial reuse possibilities exist for hazardous materials based on "Use Constituting Disposal Requirements". The material and use must meet general requirements such as use as an ingredient in manufacturing and not produce leachate levels beyond what would be produced by virgin materials. Georgia makes case by case determinations on reuse based on professional judgement and comparison of TCLP results to drinking water standards (DWS).

In Illinois, beneficial use is allowed without a permit based on the content of the waste stream and the intended use. Approval is granted on a case by case basis. Typically, approval is granted for specific uses, but approvals can be broad such as use of road base in an entire county. Three categories of steel and foundry wastes are established. Beneficially useable waste (BUW) produces a leachate with contaminate concentrations less than drinking water standards. Potentially useable waste (PUW) produces a leachate at twice drinking water standards and low risk waste (LRW) is set at roughly five times the drinking water standard. Exceptions to these general rules exist for specific leachates. Beneficially useable waste, although considered on a case by case basis, may be used as a road ballast, construction fill and, in general, as a substitute for commercially available materials including soils used for reclamation (Kauffman, 1996).

In Michigan, inert materials may be used in place of virgin material if they do not pose a greater hazard to human health or groundwater quality than the virgin material. In general, a request may be submitted to consider a low hazard waste as inert and eligible for beneficial reuse on a case by case basis. The standard of this decision is that the

material should demonstrate one of the following characteristics: hazardous content below the environmental background level or the detection limit, or less hazardous than the virgin material when bound as a component in concrete, mortar, grout, or casting molds. In Michigan, a significant amount of foundry sand is used as landfill cover, fill material, and as fine aggregate in asphalt production (Kauffman, 1996).

Generally, if WFS are not hazardous, disposal is permitted in appropriate landfills (municipal solid waste, industrial, etc.) based on a state's landfill classification system. In most cases, these classification systems appear to require a lined landfill for an industrial waste if that waste produces leachate levels above drinking water standards. Exceptions exist in some states for privately owned landfills and existing landfills with limited time before closure. Only one state, Pennsylvania, requires a double lined landfill for nonhazardous industrial waste with a minimum leachate level at 50% of the TCLP standard. (Kauffman, 1996).

While state regulatory agencies are motivated by the same goals of protecting groundwater quality and human health, the variety of standards and guidelines for tests and chemical classification thresholds in place in the states is problematic to the metal casting industry. Analytical beneficial reuse threshold levels of leachable metals and chemical compounds in industrial wastes can vary by factors of 30 or more. For example, the specified maximum leachable level for cadmium in Illinois is 0.005 mg/L as compared to Ohio where the level is 0.15 mg/L. The number of metals and chemicals possible for regulatory conformance testing can vary by a wide margin between states as well. Ohio, for instance, stipulates 10 metals and chemicals, Michigan stipulates specific test levels for 68 metals and chemicals, and Texas stipulates about 140 (Kauffman, 1996). Furthermore, there is an escalating attempt in newer regulatory policies to reduce risk by requiring more stringent testing. For instance, a 1996 Pennsylvania policy requires 'total elemental' analysis as well as 'leachate analysis' while older (pre 1996) Indiana and Ohio policies address only 'leachate levels'. At the same time the associated costs to comply with these levels of additional testing accordingly escalates, and may become so costly as to prevent the intended reuse effort (Partridge, 1997).

It is interesting to note while regulatory policies specify stringent limitations on a variety of chemical contaminants, the sampling and analysis plan submitted by the generator may not be representative of the stockpile for the intended beneficial reuse. The decision as to whether the sample is truly representative is largely left up to the generator (Partridge, 1997). In Indiana, no more than three samples are required at a collection point to characterize a waste stream, but how these samples are collected depends heavily on the discretion of the generator as to the representativeness of the sample. Even though the testing in question involves extremely sophisticated instrumentation able to detect contaminants at extraordinarily low levels, the representativeness of these results may be open to discussion. Sampling plans were primarily designed to classify a waste for disposal in permitted, protected sites, additional testing may be warranted by a reuser prior to use.

Finally, regulatory policies and guidelines do not explicitly address methodologies for considering the life-cycle issues (cost-benefit, risk, etc.) of the beneficial reuse alternatives. Although two states, Illinois and Michigan, use probabilistic risk methods, a consistent policy was not identified for guidance to regulators in comparing alternatives such as placing nonhazardous foundry sand in landfills versus a beneficial reuse activity that may result in low level, but acceptable ground water leaching concerns (Kauffmann, 1996).

In short, with states employing a wide variety of regulatory classification, test procedures, and beneficial reuse criteria for industrial, solid wastes it is understandable why WFS reuse has lagged behind industry expectations. This has been further compounded by the lack of decision based, science tools (e.g. life-cycle or risk-based analysis methods) and short and long term reuse objectives. Furthermore, test standards typically do not consider the natural presence of certain elements, in particular metals, in the native soils. As a result, a beneficial reuse material such as WFS can be subjected to more stringent test standards than natural soils.

Although state environmental regulatory agencies are the most frequently identified source of barriers to beneficial reuse of waste materials, other barriers exist. A foundry must have an organizational commitment to the reuse of WFS. Institutional barriers exist if WFS reuse is not a corporate priority because of a lack of organizational commitment to do basic tasks such as separating or screening the sand for reuse. A 'product mentality' rather than a 'waste mentality' needs to be developed. Product quality and market expansion may receive a higher priority by a foundry if spent sand, slag or dust are considered as reusable and marketable products rather than as wastes requiring disposal. Assuming that a foundry is willing to commit resources to develop a market for their WFS, product quality control is required to provide potential customers the incentive to use the material. For instance, a local concrete plant may not be willing to risk making changes from conventional fine aggregate materials if there is a perceived risk of product failure or environmental issues. The financial benefit of a low cost aggregate may not outweigh the risks in the potential user's view (Kauffmann, 1996).

3.2.5 Reuse Alternatives and State DOT Reuse Activities

As previously noted, the need for better management of solid wastes has revived recycling efforts, and has resulted in the development or consideration of a series of alternative disposal applications. The Casting Industry Suppliers Association (CISA), Sand Committee Task Force indicates the number of alternative constructive reuse options is so great that even if only 20% of the applications identified were fully developed, all of the WFS produced would be consumed. Reuse options include any practice in which sand is currently being used, including but not limited to: cement, concrete, asphalt, bricks/tile, flowable fill, geotechnical fill/roadfill, daily landfill cover, and manufactured topsoil/composting (CISA, 1995).

Towards promoting reuse efforts, CISA points out that in 1994 about 45 million Mg of sand were consumed in the manufacturing of ready mixed concrete, and using WFS in this application alone would signify a potential uptake of over 11.7 million Mg of WFS per year (CISA, 1995). However, research results using WFS in concrete have provided

conflicting results. For instance, one study looked at the reuse of WFS in concrete and concrete blocks, determining that WFS work best in applications requiring relatively fine aggregates, and when used in the appropriate proportions can yield strengths comparable to those obtained with traditional materials (McIntyre, 1992). However, another investigation into the use of WFS as a substitute for fine aggregate in concrete by the Center for By-Products Utilization (Naik, 1994) showed a substantial decrease in concrete compressive strengths likely due to binders and clays in the WFS. Additionally, the slump, which is a measure of a concrete's workability, for mixes containing WFS was considerably less, typically one-fifth the slump of mixes containing natural sands, which could manifest into placement and compaction problems.

It has also been suggested with an annual U.S. asphalt production of about 272 million Mg, if WFS were used as a fine aggregate replacement, approximately 71 million Mg of WFS could be consumed (CISA, 1995). However bituminous engineers contacted in Indiana have expressed concern over the typically smooth grain shape of WFS. WFS, in their opinion, would lack the fine aggregate angularity needed for asphalt pavement durability.

3.2.5.1 Pennsylvania

One attractive reuse opportunity for WFS is as fill or embankment material. Such applications have the ability to use large volumes of WFS and, as previously noted, the geotechnical properties of many WFS are suitable for this application. However, some foundries' disposal techniques are not currently appropriate for high-volume access (CISA, 1995), nor do the majority of foundries practice stockpiling of their sand. Consequently, their WFS are not readily accessible in the volumes needed for these applications. Some foundries have begun joint stockpiling of their WFS to provide adequate volumes for reuse applications, but have experienced quality control concerns. In an effort to provide needed volumes of WFS, two foundry-owned companies in Pennsylvania have sited and opened disposal facilities for the purpose of monofilling foundry wastes. Tipping fees charged at these landfills are \$15/Mg, significantly less than

the local municipal landfill rate of \$28/Mg. These two companies are the Process Recovery Corporation (PRC), in eastern Pennsylvania, and the Allegheny Recovery Corporation (ARC), in western Pennsylvania. The first such company (PRC), with its 33 participating foundries, has even secured a permit for the specific use of waste sand as fine aggregate in road construction. In a recent application approximately 23 Mg of WFS were utilized for road construction by the Pennsylvania Department of Transportation (PenDOT) (Bastian, 1996).

Although the ARC, which has 25 member foundries, has not been allowed similar WFS reuse, a group of 37 foundries has applied to the Pennsylvania Department of Environmental Protection (DEP) for a general WFS reuse permit. This group includes foundries associated with PRC, ARC, and others. The DEP permit, when granted, will allow much more broad use of WFS, even including use in plant nurseries. The requirements stipulated by the permit include measurement of 22 metals and 4 organics in WFS leachates. In the past, Pennsylvania foundries have found it difficult to pass leachate concentration limits for iron and manganese. Now, however, the DEP has allowed the leachate to be generated by a procedure called the Synthetic Precipitation Test rather than the TCLP; this is expected to result in lower leachate iron and manganese levels. Organics to be measured include naphthalene, benzene, toluene, and xylene, depending on the binder chemicals used. Most laboratories will test for all four due to the difficulty in specifying the exact binders used for each sample (Bastian, 1996). The DEP is reportedly ready to sign the permit application, and Pen DOT is planning a 1800 Mg WFS demonstration project. Once the permit is granted, the foundries will be faced with the logistical challenges of obtaining sufficient sand for the project and economically transporting it to the project site. Nevertheless, the granting of the general use permit is seen as a major step toward common use of WFS by the foundries in Pennsylvania (Regan, 1996).

3.2.5.2 Michigan

Placement of WFS as an embankment material was reported to be an accepted practice in Michigan, particularly around the Saginaw area, which has a considerable volume of WFS. However, this proactive stance shifted dramatically following a singular instance in which these waste sands were found to have released a phenolic leachate contaminant into adjacent waters, resulting in significant liability and cost to the Michigan DOT. This discovery actually developed as a secondary observation to a far larger, unrelated post-construction problem, but the end result was that the Michigan DOT decided to specifically preclude any future use of WFS with an appropriately written exclusive specification (Bastian, 1996).

3.2.5.3 Wisconsin

The Wisconsin DOT initially was in a 'holding pattern' regarding the reuse of WFS due to concerns regarding liability. They initially applied for a liability waiver from the Wisconsin Department of Natural Resources (DNR), but the request was disallowed. The DOT then considered pursuing an alternative course of action, whereby the DOT could establish so-called 'partnering agreements' with the involved foundries through which these parties would assume joint liability (Bastian, 1996).

The Wisconsin DOT in 1997 changed their policy regarding reuse of WFS from a "holding pattern" to a more active attempt to find reuse alternatives. The Wisconsin DOT has subsequently conducted field trials using WFS in flowable fill and as fill materials. This change in policy came after the DNR, which is responsible for regulating these wastes, determined that liability would be solely held by the waste supplier, exempting the DOT of all short or long term liabilities. The current attitude by the DOT is positive, approving the use of WFS in projects where it is suitable and economically beneficial, and where the DOT anticipates no related problems (Fister, 1997). While the Wisconsin DNR can exempt the DOT from state RCRA liability, it does not, however, have the authority to exempt it from federal CERCLA liability.

It should be noted that researchers at the University of Wisconsin at Madison have been investigating the environmental quality of WFS since the mid-1970's, soon after the United State's initial efforts at solid waste regulation. Professors Robert Ham and William Boyle have sought out opportunities to demonstrate the environmental viability of using WFS in construction projects, including a demonstration embankment project which was constructed by the Wisconsin DOT using 10,000 cubic yards of WFS. An extensive monitoring program was developed for this site, with lysimeters being installed in both the WFS and natural soil embankments to procure 'in-situ' leachates for subsequent testing, corresponding initial results proved to be extremely positive (Ham, 1993b; Bastian, 1996). Results of this study are discussed in-depth in Chapter 5.

A follow-up interim report, entitled "Evaluation of Selected Foundry Wastes for Use in Highway Construction" was presented by Lovejoy, one of Ham's co-researchers, in 1996. This report discussed the pre-construction testing of the foundry waste stream and reported on 2 years of additional environmental field testing. Solid waste streams from three Wisconsin foundries were tested for leaching characteristics using the TCLP and the American Foundrymen's Society (AFS) test. Leachates from the waste streams were found to be nonhazardous based on RCRA criteria, which agreed with earlier results (Ham, 1993b). In his conclusions, Lovejoy noted there were several parameters from the field testing which exceeded the Drinking Water Standards (DWS) at least once during the evaluation. For the WFS lysimeters, these parameters included pH, chloride and iron. For the lysimeters in the natural Wisconsin soils, the control sections, these parameters included: chromium, cadmium, sulfate, selenium, total dissolved solids (TDS), zinc, and fluoride. Groundwater monitoring wells did not show any impacts that could be attributed to either the WFS or the natural soils. The author, however, noted that the sampling time period may not have been sufficient to fully flush the WFS due to its low hydraulic conductivity.

Another use of WFS, as a fine aggregate in flowable fill, also appears promising, in part due to the smoothness of the WFS. Flowable fill is a low-strength concrete product that normally uses construction-grade sands as a raw material. Attainable characteristics

include low permeability, fluid-like consistency, and relatively fast set-up time, making it an ideal product for many applications in the utilities industry (CISA, 1995). Laboratory and field tests have indicated the use of WFS to have maintained or improved the performance of flowable fill (Stern, 1995). Although flowable fills made with WFS require more cement (due to residual binders and clays) to reach acceptable strengths, this expense may be offset by the savings when using low-cost or cost-free WFS (Bhat, 1996).

3.2.5.4 Ohio

The use of WFS in flowable fill has been studied and demonstrated in the state of Ohio since 1992. In 1994, Ohio DOT policies were revised to allow nontoxic spent foundry sand to be used in various flowable fill construction operations. In May, 1993, a demonstration and discussion forum was held at the Institute of Advanced Manufacturing Sciences in Cincinnati. At this forum, two flowable fills containing WFS were placed beside a natural sand flowable fill for comparisons. Since that time, the foundry industry and the Ohio Department of Development (ODOD) have pushed for increased education of local and city engineers regarding the use of WFS in flowable fill. In May, 1994, a large project using flowable fill made with WFS was placed at the Cuyahoga County Fairground with industry and government officials in attendance. In 1993, the Hamilton County Engineer's Office (serving the city of Cincinnati, Ohio) began requiring flowable fill for the closing of excavations made within pavement right-of-way. As part of this requirement, Hamilton County approved WFS as an aggregate in flowable fill, creating a large market for WFS reuse. The ODOD and the Ohio Cast Metal Association (OCMA) have since set goals and recommendations for increasing the usage of WFS in Ohio and for educating both end users and WFS suppliers (Stern 1995).

Ohio recently passed legislation (November, 1994) to facilitate the beneficial use of non-toxic bottom ash, fly ash, and spent foundry sand (Ohio EPA Policy 0400.007: *"Beneficial Use of Non-Toxic Bottom Ash, Fly Ash, and Spent Foundry Sand and Other Exempt Wastes"*). According to this new policy, non-toxic materials within each of these categories are exempt from regulation as hazardous or residual solid waste if they pass

certain criteria. The policy specifically states that beneficial reuse of these materials shall not require a permit by the Ohio Environmental Protection Agency. The Ohio Department of Transportation (ODOT), however, has indicated that they are “not that excited” about the prospect of reusing WFS. In the case of flowable fill, ODOT currently does not have relevant specifications to allow the use of WFS. Furthermore, current WFS do not meet ODOT’s present specifications as either a soil or granular material, and concerns were expressed about the content of silty fine material in the WFS and the resulting performance problems (Bastian, 1996).

3.2.5.5 Iowa

In Iowa, the Iowa Environmental Protection Commission passed an amendment to their administrative code entitled “Reuse of Solid Waste” (Iowa Administrative Bulletin (IAB), April, 13, 1994), which establishes their State’s seemingly progressive criteria for reuse of WFS. In short, this ruling (Amended Rule 567-108) allowed several different beneficial reuse activities without a permit for those wastes whose TCLP values were less than or equal to 90% of the Federal RCRA leachate classification limits. The proposed beneficial uses for which no permit was required included: daily cover at a landfill, road ballast, construction or architectural fill, dike or levee construction, fill base for roads, road shoulders, parking lots, or other similar uses, and any other beneficial use upon written notification by a foundry. The Iowa DOT, however, has expressed concerns about assuming liabilities as a result of using WFS. Iowa DOT specifications currently have an option for a contractor to come back with a proposal to use WFS, however, the DOT was reportedly hesitant to use these materials due to their concerns about contaminants, including metals, especially for those applications in close proximity to groundwaters. Their primary interest at the time was in using WFS in flowable fills (Bastian, 1996).

In Indiana, the Ready Mix Association has expressed concerns with hauling flowable fills containing WFS. These concerns stem around potential liability to the industry as a ‘generator’ and ‘transporter’ of a product containing a regulated material, WFS. While

such liability concerns may be minimal under state and federal RCRA regulations they remain a reality, although remote, under federal CERCLA liability provisions.

3.2.5.6 Illinois

In Illinois, the present mood about reuse of WFS by the Illinois Department of Transportation could best be described as 'vague' and 'unresolved'. As other neighboring states, this DOT carries distinct concerns about liability which might arise in the future. One unique step taken by this State was the stipulation that recyclers must furnish "Material Safety Data Sheets" (MSDS) for each group of materials which they handle. However, in the particular case of foundry operations and residuals, it does not appear that these sorts of MSDS sheets have been considered, let alone prepared, for waste foundry sands (Bastian, 1996). In turn, the prospects for using WFS by the Illinois DOT are slim at this time. Although there are permits allowing WFS reuse, there is a lack of interest in the material and the DOT has not determined whether the benefits of reuse are significant enough to warrant common application (Metcalf, 1997).

CHAPTER 4: WASTE FOUNDRY SAND LIABILITY ISSUES

4.1 Introduction

As noted in Chapter 3, according to foundries, state environmental regulatory agencies are the most frequently identified source of barriers to the beneficial reuse of WFS. While complex regulations and test procedures, which often lack consistency amongst states, create barriers for reuse of WFS, the most prevailing concern expressed by DOTs is the potential liability exposure when using a regulated waste. State regulatory agencies such as the Pennsylvania Department of Environmental Protection (DEP), Ohio EPA, Iowa Environmental Protection Commission, and the Wisconsin DNR have attempted to facilitate the beneficial reuse of WFS and in some cases reduce a reuser's potential liability under the state's solid waste regulations. However, even though a reuser may be exempt from liability under state RCRA regulations, the reuser may not be exempted from the generous liability provisions under CERCLA. The Indiana Department of Transportation (INDOT) needed an answer to this concern before allowing WFS to be used in INDOT contracts.

4.2 Potential INDOT Liability When Using Waste Foundry Sands

In the fall of 1994, INDOT asked environmental attorneys from the law firm of Stuart & Branigin to issue an opinion on INDOT's exposure (potential liability) under RCRA and/or CERCLA from the beneficial reuse of WFS. In short, their opinion (Huber, 1994) stated:

Spent foundry sands that are used as a base in road building are excluded from Indiana's RCRA regulations if the sands satisfy certain toxicity tests. As long as the sands in question satisfy these technical criteria, IDEM complies with the regulations regarding

this exclusion, and if the sands are used as a base in road building, INDOT should not incur any liability for this use under RCRA.

Even if the sands in question satisfy Indiana's RCRA exclusion, however, there does not appear to be any reason why INDOT (and possibly the generator) could not be liable under federal CERCLA if the spent foundry sands used in road construction contaminate the groundwater or otherwise become the subject of a CERCLA clean-up action. This may be unlikely, but it is a risk given the courts' current interpretation of CERCLA.

Their opinion raised, but did not address the extent to which Indiana Occupational Safety and Health Administration (IOSHA) regulations may apply to INDOT employees or contractors who are potentially exposed to regulated contaminants in WFS, during construction. IOSHA has generally adopted federal OSHA regulations regarding employees' exposure to contaminants in the work place.

The legal reasoning and case law (as of November 26, 1994) for INDOT RCRA and/or CERCLA liability using waste materials (e.g. WFS) was as follows:

RCRA generally applies to the generation, transportation, treatment, storage and disposal of 'hazardous waste' (42 U.S.C. § 9601 et seq.; Ind. Code § 13-7-8.5-1 et seq.). Indiana's regulations regarding the identification of hazardous waste generally adopt the federal rules in 40 C.F.R. part 261. 329, Indiana Administrative Code § 3.1-6-1. Under these federal rules, to be a 'hazardous waste,' a substance must first be a 'solid waste' (42 U.S.C. § 6903 (5)). A 'solid waste' is any "garbage, refuse, ...and other discarded material..." (Id. § 6903(27)). RCRA regulations define 'solid waste' as any 'discarded material' which is either:

- a) abandoned (by being disposed, burned, incinerated, or stored prior thereto), or
- b) inherently waste-like, or
- c) a secondary material (i.e. any spent material, sludge, byproduct, etc.), when recycled in any of the following four ways:
 - 1) used in any manner constituting disposal;
 - 2) burned for energy recovery used to produce a fuel or contained in fuel;

- 3) reclaimed; or
- 4) accumulated speculatively (40 C.F.R. § 26.2).

When Indiana adopted federal RCRA rules regarding the definitions of ‘solid waste’ and ‘hazardous waste’ in 1992, it retained certain exclusions relating to foundry sand in its regulations (329 Ind. Admin. Code 3.1-6-1(b)). These exclusions provide that the following activities are excluded from Indiana’s solid waste RCRA program:

- (14) The legitimate use of foundry sand which has been demonstrated as suitable for restricted waste site Type III under the provisions of 329 IAC 10-3-1 including the use as a base for road building, but not including use for land reclamation except as allowed under subdivision (15).
- (15) Other uses of solid waste may be approved by the commissioner if the commissioner determines them to be legitimate uses that do not pose a threat to public health and the environment. (329 Ind. Admin. Code 10-3-1(14) & (15)).

For WFS to satisfy this exclusion, it must satisfy the technical criteria in 329 Ind. Code 10-9-4, regarding foundry waste disposed of at a restricted waste site, Type III. Testing requirements include the TCLP test (chemical analysis of leachate produced from the TCLP leaching procedure) for arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver and, among other things, a neutral leachate test for other parameters such as chlorides, phenols, iron, etc. Waste foundry sands that satisfy this exclusion are not subject to Indiana’s RCRA program if they are used for a ‘legitimate use’, such as a base for road building (Id. 10-3-1(14)). In addition to the roadbase definition, INDOT must satisfy environmental siting criteria consistent with the siting criteria for the restricted waste type (329 IAC 10-33-1 (a) (1-7)).

Even if INDOT’s use of WFS is exempt from Indiana’s RCRA program because the sands are ‘non-toxic’ and are used as a base in road construction, the question remains whether liability under CERCLA may apply. CERCLA, as well as Indiana’s mini- ‘Superfund statute’, (Ind. Code § 134-7-8.7-1 et seq.) authorizes the government (and private parties in

some instances) to take action in response to the “release or substantial threat of release” of any ‘hazardous substance’ regardless of the effect of the release on the environment, or of “any pollutant or contaminant which may present an imminent and substantial danger to the public health or welfare” (42 U.S.C. 9604 (a)(1)). ‘Hazardous substances’ include RCRA ‘hazardous wastes’ and numerous other listed chemicals (Id § 9601 (14)).

Liability under CERCLA will not lie unless 1) the site is a ‘facility’, 2) a ‘release’ or a ‘threatened release’ of a ‘hazardous substance’ has occurred or is occurring, 3) the release or threatened release has caused response costs to be incurred, and 4) the defendant falls within one of the classes of responsible persons listed in CERCLA § 107 (a), 42 U.S.C. 9607(a). Kerr-McGee Chemical Corp. v. Lefton Iron & Metals Co. (14 F.3d 321, 325 (7th Cir. 1994)).

‘Facility’ includes any site or area where a hazardous substance is located (42 U.S.C. § 9601(9)). The universe of potentially responsible parties includes government as well as private persons who are 1) owners or operators of a facility, 2) persons who owned or operated the facility at which the hazardous substances were disposed at the time of disposal, 3) persons who by contract or otherwise arrange for the disposal of hazardous substances (e.g. ‘arrangers’ or ‘generators’), and 4) persons who accept hazardous substances for transport to disposal or treatment facilities from which there is a release or threatened release (‘transporters’) (42 U.S.C. § 9607 (a)).

A letter from the Ohio Cast Metals Association dated November 19, 1993, suggests that INDOT arguably should be able to avoid CERCLA liability because the foundry sands are being reused such that they are not ‘discarded’, ‘disposed of’, or ‘abandoned.’ Stated differently, this argument is that the sands in question are not a ‘waste’. Under CERCLA, ‘disposal’ has the same meaning as under federal RCRA (Id § 9601(29)). There, ‘disposal’ references the term ‘waste’ (Id. § 6903(3)). The courts that have addressed such arguments, such as in Catellus Development Corp. v. United States (34 F.3d 748 (9th Cir. 1994)), and have concluded that case law regarding the definition of ‘disposal’, ‘discarded material,’ ‘waste’, etc. under RCRA applies to CERCLA cases as well. The Court in

Catellus relied heavily on cases construing RCRA's definition of 'disposal' in holding that a party who sold spent automotive batteries to a lead reclamation plant could be liable under CERCLA as one who 'arranges' for the disposal of a hazardous substance (34 F.3d at 752-52).

A series of cases have analyzed the meaning of 'discarded material', 'disposal', and 'waste' under RCRA. First, in American Mining Congress v. United States, ('AMC I') (824 F.2d 1177 (D.C. Cir. 1987)), the court addressed whether Congress used the term 'discarded' in RCRA in its ordinary sense -- 'disposed of' or 'abandoned' -- or whether it used this term more broadly to encompass materials no longer useful in their original capacity, though destined for immediate reuse in another phase of an ongoing production process (824 F.2d at 1185). In AMC I, the court concluded that Congress used the term in the former manner, and held that only materials that are 'disposed of' or 'abandoned' can be regulated under RCRA (Id at 1186).

Next, the court in American Petroleum Institute v. United States (906 F.2d 729 (D.C. Cir. 1990)), expanded the definition of 'discarded material.' There, U.S. EPA maintained that the holding in AMC I precluded it from regulating as RCRA hazardous waste certain slag materials if these materials were to be delivered to a plant from metal reclamation. The American Petroleum court disagreed, maintaining:

The issue in [AMC I] was whether the EPA could, under the RCRA, treat as 'solid wastes' "materials that are recycled and reused in an ongoing manufacturing or industrial process." We held that it could not because "[t]hese materials have not yet become part of the waste disposal problem; rather, they are destined for beneficial reuse or recycling in a continuous process by the generating industry itself." Materials subject to such a process were not 'discarded' because they were never "disposed of, abandoned, or thrown away."....Unlike the materials in question in AMC I, [the slag in question] is indisputably 'discarded' before being subject to metals reclamation. Consequently, it has "become part of the waste disposal problem" American Petroleum (906 F.2d at 741).

Even though this reading of the AMC I case was not entirely accurate, the American Petroleum court held that the slag material at issue could be regulated under RCRA because it was ‘discarded’ or ‘abandoned’ by the industry for a time before it was delivered to the plant for metal reclamation.

Later, in American Mining Congress v. United States (‘AMC II’), (907 F.2d 1179 (D.C. Cir. 1990)) the court again expanded the definition of ‘solid waste.’ There, the petitioners, in reliance on AMC I, argued that three types of materials at issue were not ‘solid wastes’ because the sludge that contained these materials were stored in surface impoundment’s for a time before they were to be reclaimed. The AMC II court rejected the petitioners’ reading of AMC I, explaining:

Petitioners read AMC I too broadly. AMC I’s holding concerned only materials that are “destined for immediate reuse in another phase of the industry’s ongoing production process,” and that “have not yet become part of the waste disposal problem”. Nothing in AMC I prevents the agency [U.S. EPA] from treating as ‘discarded’ the wastes at issue in this case, which are managed in land disposal units that are part of wastewater treatment systems, which have therefore become “part of the waste disposal problem,” and which are not part of the ongoing industrial processes (AMC II, 907 F.2d at 1186).

The same issue arose again in United States v. ILCO, Inc., (996 F.2d 1126 (11th Cir. 1993)). There, ILCO purchased spent batteries from various sources and then recycled them. It contended that, because it recycled the spent batteries, the batteries had not been ‘discarded’ and were therefore not a solid waste. The ILCO court disagreed, insisting that although the lead components in the spent batteries were valuable for ILCO’s purposes, these materials were ‘discarded solid waste’ because someone up-stream had discarded the battery in which these components were found. The fact that someone had discarded the battery up-stream “does not change just because a reclaimer has purchased or finds value in the components” (966 F.2d at 1131).

The Fourth Circuit addressed this issue again in Owen Electric Steel Co. v. Browner (F.3d, 1994 WL 554,656 (4th Cir. October 12, 1994)). Owen produced steel in an electric

arc furnace. During production, crushed limestone was added to the furnace to remove certain non-ferrous constituents from the molten metal. In this process, the non-ferrous materials combined with the limestone, creating slag material containing trace amounts of metallic oxides. After the slag floated to the surface of the molten metal, Owen would remove it and place it in holding bays. The slag generally would lie in these holding bays for approximately six months on bare soil where it would cure. After the slag cured, Owen sold the slag to the construction industry for use as a road base material and other commercial purposes (1994, WL 554,656, at *1).

After reviewing the relevant cases, the Owen Electric court concluded that the “fundamental inquiry in determining whether a byproduct has been ‘discarded’ is whether the byproduct is immediately recycled for use in the same industry; if not, then the byproduct is justifiably seen as ‘part of the waste disposal problem,’ and therefore as a ‘solid waste’” (Id. at *4). Because Owen’s slag was not immediately used in Owen’s production process, and was left untouched in holding bays for approximately six months before it was sold, the court concluded that “it cannot be said that the material was never ‘disposed of, abandoned or thrown away’” (American Petroleum, 906 F.2d at 741). As a result, even though Owen ultimately sold its slag to others for use in roadbed construction, the court held that Owen’s slag was subject to RCRA because it was not “destined for beneficial reuse or recycling in a continuous process by the generating industry itself.”

According to the courts in Owen Electric, ILCO, AMC II, American Petroleum, and Catellus Development, whether a material is ‘discarded’ or ‘disposed of’ for purposes of federal RCRA (and by definition CERCLA) turns on whether the material in question is immediately used or recycled in a continuing process by the generating industry. Under this test, the foundry sands INDOT would acquire from foundries are almost certainly ‘disposed of’ for purposes of CERCLA. Although INDOT may be able to distinguish cases like Owen Electric, somewhat, if there is no need for the foundry sands to cure for six months or so, the courts’ reasoning suggests that even if INDOT reused the sand

immediately they will be considered discarded because the 'reuse' is by someone other than the foundries that generated the sands.

Consequently, INDOT probably could not argue that the foundry sands are waste vis-à-vis the foundries and non-waste for INDOT's purposes. Although most of the cases addressing this question approach the problem from the perspective of the industry, or 'seller' of the material, it is clear from the decision in ILCO (966 F.2d 1126), that this makes no difference in terms of INDOT's exposure as an 'end user.' In ILCO, even though the buyer or user of the spent batteries recycled them to obtain valuable lead components from the batteries, the court concluded that the batteries were 'discarded solid waste' because someone up stream had discarded the batteries (996 F.2d at 1131). As a result, according to the ILCO court, even though INDOT may be obtaining the foundry sands for beneficial purposes, this does not change the status of the sands as 'discarded material' or a 'disposed of' waste for purposes of the analysis.

As such, the foundry sands would be subject to CERCLA to the extent that they are a 'hazardous substance.' A 'hazardous substance' includes RCRA 'hazardous waste' and, among other things, any "imminently hazardous chemical substance or mixture" that is the subject of a clean up action under CERCLA, but it does not include any waste that has been excluded from federal RCRA by an "Act of Congress" (42 U.S.C. § 9601(14); 40 C.F.R. § 302.4 (listing numerous regulated hazardous substances)). The question this definition raises is whether foundry sands that satisfy Indiana's RCRA exclusion can be regulated as a 'hazardous substance' under federal CERCLA.

U.S. EPA has not taken a position on this question (at least not one that is published) and there does not appear to be any reported case law on point (as of November 26, 1994). However, cases regarding certain slag and ash mining wastes (known as 'Bevill wastes'), which have been exempted from federal RCRA by Congress, suggest that foundry sands probably can be the subject of a CERCLA clean up action as a hazardous substance, even though they are excluded from Indiana's RCRA program. For instance, the courts in Louisiana-Pacific Corp. v. ASARCO, Inc. (24 F.3d 1565, 1572-1575 (9th Cir. 1994)) and

Eagle-Picher Industries v. U. S. EPA, (759 F.2d 922 (D.C. 1985)), which appear to represent the majority rule, held that Bevill wastes are subject to CERCLA if they contained other regulated hazardous substances, such as copper, lead, arsenic, and zinc. The ASARCO court also held that the ‘waste’ in question could simultaneously be a useful ‘product’ for purposes of state laws and a hazardous substance for purposes of CERCLA (24 F.3d at 1575). United States v. Iron Mountain Mines, Inc. (812 F. Supp. 1528 (E.D. Cal. 1992)), an earlier case that reached the opposite conclusion, was implicitly overruled by ASARCO.

These cases thus suggest that waste foundry sands can be regulated under CERCLA as a hazardous substance, even if they satisfy Indiana’s RCRA exclusion. Testing performed by Purdue University on behalf of INDOT have shown waste foundry sands may, in certain instances, contain fluorides and organic compounds such as coal tar derivatives and possibly other contaminants that may present environmental complications. Any such sands would probably contain regulated CERCLA hazardous substances and could therefore be the subject of a subsequent clean up action for which INDOT could be liable.

Under CERCLA, there does not appear to be any legitimate reason why INDOT could not be liable for clean up costs if the foundry sands in question are the subject of a subsequent clean up action. According to the leading cases analyzing what constitutes ‘discarded material’ for purposes of RCRA and, by definition, ‘disposal’ of a ‘waste’ under CERCLA, INDOT’s use of waste ferrous foundry sands as a base in road construction probably constitutes ‘disposal’ or a ‘solid waste’. If these sands exhibit characteristics of a hazardous waste or contain hazardous substances or otherwise pose a threat to the environment, these sands could be deemed a ‘hazardous substance’ for purposes of CERCLA. There is, therefore, some risk that INDOT and the generators of the foundry sands could expose themselves to liability under CERCLA even if the foundry sands are exempt from Indiana’s RCRA provisions.

Because CERCLA is a federal statute, there probably is no state legislative solution to this problem. To the extent that the WFS satisfy Indiana’s RCRA exclusion, it would seem to

be unlikely that they would pose a serious threat to the environment or otherwise become the subject of a CERCLA clean up action. However, this is a technical question which one should weigh carefully in view of CERCLA's expansive definition of 'hazardous substance'. At the present time, there does not appear to be any legal reason why INDOT (and possibly the generators) could not be liable under CERCLA if INDOT uses WFS as a base in road building and these sands contaminate groundwater or otherwise threaten the environment.

4.3 Liability Under CERCLA (Superfund)

Liability under Superfund is very broad in its scope of enforcement and in an effort to find deep pockets to pay for remediation costs, administrators and judges have had incentives to further broaden Superfund's liability. Liability under Superfund is retroactive so as to include responsible persons dating back to the origination of the released contaminants. Furthermore, substances that were not deemed hazardous in the past, based on chemicals and/or concentrations may be regulated as hazardous today, resulting in subsequent liability. Furthermore, 'proper disposal methods' in the past may be deemed unacceptable by today's standards, again resulting in subsequent liability. Superfund was never construed to be fair.

Liability under Superfund is also strict liability, i.e. liability is not based on fault and is supported by ample case law (e.g. United States v. Tex-Tow, Inc., Colorado v. Asarco, Inc., United States v. Wade,). The end result is that the release need not be of a particular defendant's waste for that defendant to be liable.

Finally, as a general rule (subject to certain exceptions), liability under Superfund is joint and several, meaning each potentially responsible party (PRP) can be liable for all the costs incurred in a clean up. While CERCLA does not explicitly call for joint and several liability, some courts have interpreted the statute to impose this liability on PRPs.

Liability under CERCLA is very broad resulting in ample opportunity for litigation. Consequently, in many cases only a small percentage of funds collected and authorized under Superfund have actually been used for site remediation, with the majority of funds going toward legal fees. Businesses have had incentives to litigate in the long term hoping for more fairness in judicial judgments, as well as eventual legislative reform. In short, Superfund, which was envisioned by legislators to be the ‘large solution’ to the problem of cleaning up hazardous waste sites, has resulted in continuing litigation problems, resulted in relatively little actual site remediation, and can inhibit environmentally friendly programs such as the recycling and reuse of waste materials.

4.4 CERCLA (Superfund) Liability Reform

CERCLA and SARA (Superfund Authorization, Reauthorization Act) liability could likely be reformed to promote and not discourage the reuse of nonhazardous waste streams. Superfund liability reforms were contained in the proposed bills HR 2500 IH and SB 1285, Reform of Superfund Act of 1995 during the 104th U.S. Congress. While a complete review of the proposed liability reforms is beyond the scope of this paper, it is noteworthy Section 215, Title II, Clarification of Liability for Recycling Transactions within HR 2500 attempted to address liability associated with recycling of ‘recyclable materials.’ For instance, “a person who arranged for the recycling of recyclable material shall not be liable.” “Recyclable material means scrap paper, scrap plastic, scrap glass, scrap textiles, scrap rubber (other than whole tires), scrap metal, or spent lead-acid, spent nickel cadmium and other spent batteries...” Unfortunately, waste streams such as WFS were not mentioned. Furthermore, HR 2500 generally attempted to address potential ‘hazardous’ material contamination of recyclables with wording such as “minor amounts of material, reasonable care,” and exclusion (i.e. therefore, liable) if “the person added hazardous substances to the recyclable material for purposes other than processing for recycling...” Interpretation of such language, however, would likely be evolved by administrators and the courts, possibly leading to further litigation and a continued reluctance to use recyclables as well as other waste streams. In lieu of, or perhaps in addition to such reforms, a statistically based testing protocol (which also addresses siting

criteria, risks, etc.) could be considered which a waste (or recyclable material) would have to pass prior to use. Once passed, the suitability of the material would be established and the user exempted from further concerns of liability. In short, Congress could reform Superfund's expansive liability provisions by replacing the current subjective interpretation of Superfund's intent with the more objective criteria of a total testing protocol (TTP).

It may be argued that no testing protocol is completely accurate, all of the time. Consequently, a 'hazardous' substance release could occur as a result of the 'appropriate' use of a waste after passing a TTP, although this type of release would likely be the exception. Furthermore, generators may be willing to pay a surcharge to fund a 'cleanup' in return for avoiding costly landfill tipping fees. Even environmentally conscious reusers may be willing to pay a surcharge for the opportunity to use a waste material in lieu of virgin materials as long as the costs remained relatively comparable. Unfortunately, Superfund liability reform has not been passed by Congress and the spectre of potential liability remains a primary concern with reusers of WFS.

Another look at the economic reality of waste disposal issues may also be needed. There is a concern of regulating 'wastes' to such extremes, in terms of obviating risk, that the survival of the industries becomes an issue. As a case in point, coke production in the United States is so tightly regulated that it is actually cheaper, in some instances for the raw coal to be shipped overseas, coked in foreign plants, and then shipped backed to domestic users. Understandably, there is a valid argument for continued source reduction and striving to make wastes continually 'cleaner'. However, some wastes will likely persist as long as an industry exists and past testing of WFS, for instance, has shown many WFS which could beneficially be used in roadway construction projects at a savings to taxpayers is instead being landfilled as a result of liability concerns stemming from current regulations.

Furthermore, it appears the media and others have, at times, potentially over-dramatized the severity of environmental problems, to the point where the public has lost a realistic

perspective in terms of risk. For instance, the public may falsely believe that Congress' environmental advocacy can provide them with regulations which will do away with risk all together. In turn, the public has come to assume that risk levels for any reuse project would have been completely eliminated by excessive up-front testing not realizing that the associated costs may prevent the reuse all together. During the INDOT WFS reuse project on CR 206, INDOT researchers repeatedly were called by the local public indicting the WFS "used to be toxic," when in fact the Auburn WFS was one of the 'cleanest' sands tested in the state. Public misinformation of this sort can quickly create a negative mood towards reuse applications.

CHAPTER 5: ENVIRONMENTAL CHARACTERIZATION OF WASTE FOUNDRY SAND

5.1 Prior Chemical Characterization of Foundry Wastes

In an effort to further understand the potential release of contaminants present in foundry wastes to the environment, research groups have initiated chemical analysis of WFS leachates, lysimeter samples, and groundwater samples. In general, the list of chemicals found present in WFS, at significant concentrations, vary and include both regulated as well as unregulated contaminants. Furthermore, measured concentrations depend on the leaching procedure used as well as the waste source (Bastian, 1996).

In the late 1970's, prior to the development of the EP Tox and TCLP tests, researchers from the University of Wisconsin at Madison developed a shake flask procedure for leaching spent foundry sands and subsequently measured organic carbon, chemical oxygen demand (COD), phenol, cyanide, fluoride, sulfates, and pH in leachates from wastes from several ferrous foundries (Boyle and Ham, 1979). For some of these tests, the component parts (i.e., system sand, core butts, core room sweepings, slag, dust collector discharge, scrubber discharge, refractors, and cleaning room wastes) of these wastes were tested as well as the composite waste stream. It was seen that COD measurements in the separate constituents provided a reasonable estimate of COD concentrations in the mixed wastes; phenol levels, however, were often much higher in the composite wastes than was predicted by phenol release by the individual constituents. Laboratory lysimeter studies based on actual rainfall amounts yielded maximum concentrations of 14-120 mg/l organic carbon; 75-290 mg/l COD; 25-400 ug/l phenol; 80 ug/l cyanide; 3-120 mg/l fluoride; 30-1,220 mg/l sulfates; and pH ranges of 7.2 to 10.0 in leachates from the three ferrous sands studied. Phenol and COD levels were lower than

those observed in the shake flask tests. In shake flask and lysimeter testing, metal concentrations were seen to be very low (Bastian, 1996).

A subsequent study by the same group (Ham et al., 1981) in which sands were combined in the laboratory to simulate a WFS's waste stream after being exposed to different temperatures and various metal pours reported the concentrations of metals released. The concentrations detected were the cumulative results of three elution's using the shake flask procedure. The measured values were low and no trends were observed between metal release and level of temperature exposure.

In the mid 1980s, researchers from this group, in conjunction with RMT Inc., an environmental engineering consulting firm in Wisconsin, continued their evaluation of WFS (Blaha, 1986). Analysis of leachates of WFS from the landfills of several ferrous foundries were compared with analysis of lysimeter leachates, as well as groundwater samples, from the areas surrounding the landfills. Both the EP Tox and EP water leaching tests were used. None of these wastes were found to be toxic when leached according to the EP Tox protocol, meaning, for example, no unsaturated zone sample exceeded 100 times the NPDWS for any regulated constituent concentration. A few parameter concentrations in the WFS' leachates did exceed drinking water standards (1986) of which manganese, iron, and fluoride were the most common. The analysis of a segregated waste from one of the foundries indicated that the exclusion of a waste fraction from a particular source in the process could greatly reduce total concentrations of certain parameters. The example given indicated that the cleaning room dust, which is only 2.5% of the total waste, contributed 88.8% of the leachable iron, 33.8% of the leachable manganese, and 35.5% of the leachable fluoride, as compared to the measured values obtained from the mixed waste analysis (Blaha, 1986).

The primary drinking water parameters detected in the lysimeter samples were cadmium, chromium, lead, and silver, as well as fluoride, with a few cases exceeding drinking water standards (Blaha, 1986). The groundwater results were presented as an evaluation of the impact of the landfills on the groundwater by comparing up- and down-gradient sample

constituent concentrations. Groundwater analysis indicated no drinking water standard violations of primary metal concentrations except for barium and mercury, which were detected in the groundwater around one landfill at levels near or just above the drinking water standards. Concentrations of several secondary drinking water parameters were suspected to have increased as a result of exposure to the site; these included iron, manganese, TDS, sulfate, fluoride, and chloride. These increases were not considered alarming as these chemicals are commonly found in shallow groundwater at concentrations exceeding the drinking water standards, which was the case with some of the up-gradient well samples analyzed in the study (Salazar, 1997).

The researchers indicated there was no conclusive method of correlating laboratory leachate concentrations with lysimeter or groundwater contamination. The EP Tox and EP water leaching tests paralleled lysimeter results, but at lower parameter concentrations than found in the lysimeter samples. Large variations were observed between lysimeter and groundwater samples, and between samples from the same source taken at different times. A summary of the reported measured parameter concentrations for lysimeter and EP Tox leachate samples is shown in Table 5. The actual leachate values were not reported but were calculated using the given lysimeter results and a listing of the fraction-difference between lysimeter results and EP Tox results (Salazar, 1997). While the researchers indicated all values to be below 100 times the drinking water criteria, calculations indicated that iron and manganese exceeded this criterion one time each. However, regulations require these parameters be measured using the EP water test. The sample in which iron exceeded the 100 times criterion, when leached using the EP water test, yielded no detectable iron concentration. For the sample which exceeded the manganese concentration criterion, the value detected using the EP water test was not given. It should be noted that iron and manganese are secondary drinking water parameters and are regulated not for health concerns, but rather for aesthetic (taste or color) effects (Salazar, 1997).

Table 5: Lysimeter and Leachate Parameter Concentration Ranges from Ferrous Foundry Landfills (adapted from Blaha, 1986)

Parameter	Drinking Water Standard (as of 1986)	Detection Limit	Lysimeter Concentration Range	Leachate Concentration Range (estimated)
Arsenic ⁺	0.05	0.005	BDL	BDL
Barium ⁺	1.000	0.46	BDL	BDL
Boron	1.000	0.7	BDL	BDL
Cadmium ⁺	0.010	0.001	BDL	0.002-0.05
Chromium ⁺	0.050	0.003	0.1	0.001-0.04
Copper	1.000	0.002	0.007-0.084	0.001-0.008
Iron	0.300	0.004	0.14-1.40	0.002-71**
Lead ⁺	0.050	0.10	0.06	0.005-0.03
Manganese	0.050	0.002	0.05-1.10	0.93-6.5**
Mercury ⁺	0.002	-	<0.0029-<0.0085	-
Selenium ⁺	0.010	0.130	BDL	BDL
Silver ⁺	0.050	0.002	BDL	BDL
Zinc	5.000	0.001	0.056-0.28	0.28-5.4
TOC	-	-	13-105	-
Phenols	0.300	0.05	BDL	0.08
Cyanide	0.200	0.02	0.06-0.10	-
Fluoride	1.400-2.400	0.4	0.5-9.6	0.4-16
pH	-	-	7.1-8.2	-

All units in mg/l except pH

BDL: Below Detection Limit

⁺ Primary Metals

** Values Exceed 100 times Drinking Water Standard

Another study by the same researchers from the University of Wisconsin and RMT Inc. (Krueger, 1989) looked at the variability of leaching characteristics of ferrous foundry wastes. As in their previous study, these wastes were determined to be non-toxic according to the EPA extraction procedures. The analyzed constituents were cadmium, chromium, copper, iron, lead, manganese, zinc, cyanide, phenol, and fluoride. Results indicated that analytical procedures introduced a small variability (average variation was 6.4%), while sample handling and preparation introduced a potentially large error (average variation was 45%). Variability contribution differences between EP Tox and EP water tests were similar. Sample variation over time indicated significant variations with averages around 50% for measured parameters in baghouse dust, core butts, and

system sand. Except for copper, lead, and phenols all measured leachate concentrations reported were within the ranges measured by this group earlier, as listed in Table 5 (Salazar, 1997).

Copper had a concentration range of 0.014-0.195 mg/l, lead a baghouse dust concentration of 0.087 mg/l, and phenol a baghouse dust concentration of 0.121 mg/l. Of these values, only the lead concentration exceeded the drinking water standard (1986). The lead detection limit was only 0.1 mg/l, and all samples were below the detection limit; therefore the given value was calculated using probability curves and should be used with caution (Krueger, 1989).

Organics were also measured in groundwater collected at four Wisconsin landfills containing only ferrous foundry wastes (Krueger, 1989). No organic species was observed to have a concentration above the quantitation limit; several compounds at trace levels were tentatively identified. These included tetrachloroethene, naphthalene, chloroform, 1,1,1-trichloroethane, 1,2-dichloroethane, and di- and trichloroethene. It was suggested that some of these findings may have been due to laboratory error. As in previous work (Blaha, 1986), concentrations of groundwater contaminants did not correlate well with laboratory leachate test concentrations. A later study by members of this research group conducted an evaluation of the potential for leaching of organics from WFS (Ham, 1993a). Nine binder and core making processes, representing the major processes used by the casting industry, were chosen, including: phenol formaldehyde, phenolic urethane, furan hot box, furan no-bake, phenolic ester, core oil, phenolic isocyanate, alkyd isocyanate, and furan warm box. Foundry wastes examined which would likely contain organics included excess system sand, core butts, and core room sweepings. The TCLP leaching procedure was used for toxicity characterization. Most of the organics identified by gas chromatography/mass spectroscopy (GC/MS) analysis were quantified using gas chromatography with a flame ionization detector (GC/FID).

Organic chemicals which were detected were deemed worthy of comment if they were included on one of four regulatory lists promulgated by EPA. These were (1) the priority

pollutant list, which identifies chemicals which are environmental hazards and are found in water (88 compounds, excluding pesticides and PCBs); (2) the TCLP chemical list (38 compounds, excluding pesticides); (3) drinking water standards; and (4) a list included with the proposed solid waste disposal facility criteria under Subtitle D of the Resource Conservation and Recovery Act (RCRA), intended to apply to the release of hazardous constituents from nonhazardous waste disposal facilities. The chemicals which were detected in leachates from any of the nine foundry waste types and were included on any of these lists are included in Table 6 (Bastian, 1996).

**Table 6: Organic Chemicals Measured in Waste Foundry Sands
(Ham, 1993a)**

Chemical Compound	Quantitation Limit Limit (ppb)	Maximum Concentration (ppb)
Acetone	100	200
Benzene	2	11
Benzoic Acid	N.D.	400
2,4-Dimethylphenol	20	120
Ethylbenzene	0.4	24
1,1,1-Trichloroethane	2	49
Naphthalene	1	480
2-Methylnaphthalene	1	320
Phenol	30	540
Dimethylphthalate	40	61
Phenanthrene	30	38
Tetrachloroethene	2	7
Toluene	0.5	61
Cresols	30	150
Xylenes	0.4	140

As shown by the data, no organic chemical was found at a concentration of above 1 ppm, nor were any of the chemicals found at concentrations high enough to be considered hazardous. The only chemicals exceeding the standards set in any of the above regulatory lists were benzene, which was found at concentrations higher than the DWS maximum contaminant level (MCL) in leachates from three sands, and tetrachloroethene, which was found at a concentration equaling a 'trigger level', calculated to be used where

DWS have not been established. Both of these standards, however, were designed to be applied to groundwater or drinking water rather than applied directly to leachate; it would be unlikely that concentrations of these compounds in groundwater would ever reach the above mentioned standard levels. Core oil and phenolic urethane binder systems leached the greatest number of organic chemicals (Ham, 1993a).

A three-year study (previously referenced in Chapter 3) on contamination stemming from the use of WFS was performed on Wisconsin WFS and soils by the Wisconsin-Madison research group in the early 1990's (Ham, 1993b). WFS from three foundries were used to generate leachate according to TCLP and American Foundrymen's Society (AFS) Laboratory Leach Test protocols. Characterization of these leachates included testing for twelve metals, eight inorganic chemical groups, and bulk parameters including alkalinity, conductance, hardness, pH, total dissolved solids (TDS), and total organic carbon (TOC). The results from the sands were compared with soils considered typical of agricultural or construction soils. None of the sand or soil samples were hazardous according the RCRA criteria. Causes of potential adverse impacts were classified into two groups: top priority parameters, which had average values significantly greater in WFS leachates than in virgin soils and at least one sample which exceeded drinking water standards (DWS); and second priority parameters, which met one but not both of the above conditions. The top priority parameter of greatest concern was iron, which was found at concentrations higher than the DWS in TCLP leachates from all three foundries and in an AFS test leachate from one of the foundries. Additional top priority parameters included fluoride, for which the secondary DWS were exceeded, pH, and TDS. Second priority parameters included arsenic, chromium, copper, manganese, zinc, phenolics, and sulfates (Bastian, 1996).

A field study was then designed and implemented, in which groundwater and leachate samples were collected from test piles throughout a one and one-half year period. The amount of leachate generated was small due to the low permeabilities of the WFS piles. Arsenic was found at concentrations above the DWS in leachate from WFS and natural soil piles. Chloride and pH exceeded DWS in WFS leachate on one date each;

chromium, cadmium, and lead exceeded the DWS in leachates from natural soil piles only. Iron exceeded the DWS in only one WFS leachate. Based on these data, the researchers concluded that the WFS used in this study performed comparably to natural soils (Ham, 1993b).

A study conducted at Purdue University (Javed, 1993) presented constituent concentrations obtained from leachates of various green sand process sands using the EP Tox test and EP Water test used for solid waste classification in Indiana. The results, presented in Table 7, indicate these wastes are non-hazardous according to RCRA criteria. Some values for the primary metals were obtained using the TCLP test, instead of the EP Tox test as required by Indiana regulations. Parameters tested using the EP water test were generally below IDEM's Type IV classification, except for some fluoride, pH and zinc values. These parameter concentrations, except for some zinc values, were well below Type III classification levels.

5.2 Bioassay Characterization of Waste Foundry Sand

5.2.1 Introduction

Chemical-specific leachate testing, as covered in previous sections, has several advantages, including the ability to measure very low levels of contaminants; the opportunity to utilize well-understood and accepted protocols; and acceptable comparisons with standards, other wastes, and even unrelated risk factors. It is particularly applicable to industrial wastes whose constituents are known and which vary little over time (Bastian, 1996). Exhaustive chemical specific testing of WFS, however, can become expensive and as previously noted preclude the intended beneficial reuse effort.

Table 7: Maximum Contaminant Concentrations for WFS Samples for Four Green Sand Molding Processes (Javed, 1993)

1) Parameters Using the EP Tox Test

Parameter	Maximum Concentration (mg/L)
Arsenic	0.02
Barium	0.50
Cadmium	0.01
Chromium	<0.05
Lead	0.09
Mercury	0.002
Selenium	0.03
Silver	<0.05

2) Parameters Using EP Water Test

Parameter	Maximum Concentration (mg/L)
Barium	<0.5
Boron	0.4
Chlorides	<100.0
Copper	0.12
Cyanide, total	<0.06
Fluoride	1.59
Iron	1.06
Manganese	<0.05
Nickel	<0.1
Phenols	<0.25
Sodium	35.98
Sulfate	85.03
Sulfide, total	<1.00
TDS	236.0
Zinc	60.0
pH	7.8-9.8

When measuring the toxicity of samples which are not well characterized, another method is sometimes used: bioassay testing. Simply put, a bioassay is a test which directly measures the effect of a particular environment on a living organism. Ideally, the effects on the organisms can be correlated with risks to humans or other environmentally sensitive species more effectively than can simple concentrations of contaminants. This is partly due to the fact that in contaminated environments, more than one chemical

compound or species is often present. The effects of the combined toxicants on living organisms are not necessarily simply additive (Bastian, 1996). Toxicity can be antagonistic, or less than the sum of the individual toxic effects; additive; synergistic, or greater than the sum of the individual toxicities; or neutral, in which the effect of one or more species is masked or negated by the presence of other compounds (Dutka and Kwan, 1982). Interaction between contaminants or other environmental variables cannot generally be predicted *a priori*. Thus, an advantage of a bioassay is that this interaction can be detected.

Bioassays can be separated into acute tests and chronic tests. An acute test measures the response of the organism over a short period of time—usually a few minutes to a few hours. The response measured is usually death or inactivation of the organism. Chronic tests, on the other hand, measure the effects of an organism's environment over a period comparable to its lifetime. Detection of cancers, mutations, or changes in the organism's reproductive abilities can be used as endpoints for chronic toxicity tests. Chronic tests are generally more sensitive to environmental pollutants, but are much more costly, time-consuming, and labor-intensive (Bastian, 1996).

Bioassays have been used for many different applications. For example, a wide variety of products manufactured for human use and consumption (e.g., cosmetics, pharmaceuticals, etc.) are routinely pre-tested and certified to obviate any harmful impact on humans, in terms allergenic response, dermatitis, etc. Bioassay testing has also become a commonplace requirement for qualifying the acceptability of wastewater effluents based on a so-called 'whole effluent toxicity' (WET) analysis. Bioassay testing has become a standard requirement for most wastewater treatment operations, wherein these effluents must be proven to be suitably non-toxic for their permitted discharge. These bioassay requirements, as are included within a facility's National Pollutant Discharge Elimination System (NPDES) permit. This requirement was a result of the EPA recognizing a need to restrict and control the discharge of toxins as a whole...and that effluent limitations on specific compounds would not necessarily provide an adequate measure of protection. Their implicit concern was that the chemical-specific

requirements would not provide such safeguards against unregulated toxins or for those compounds whose impacts are additive, synergistic, or antagonistic (Bastian, 1996).

Several different types of bioassays have subsequently been developed, with acute and chronic fish and invertebrate options most commonly being stipulated. The resultant expectation is that the permitted effluent should not exhibit a toxic response during its bioassay evaluation at levels of dilution commensurate with local conditions under worst-case conditions (i.e., with a permissible dilution level of one-quarter the receiving water body's 10-year low flow value). In the event that a toxic condition is consequently identified, these facilities are then tasked with the responsibility to perform 'toxicity reduction evaluations' (TREs) in order to identify the culprit compounds. The responsible chemical(s) can then assumedly be traced back to the originating source and subsequently eliminated by appropriate enforcement action (Bastian, 1996).

Extending beyond wastewater operations, environmental bioassay testing has recently assumed a considerable measure of interest...although its actual regulatory application has not yet become commonplace. Bioassay measurements of sediment toxicity likely represents the closest analogy to the sort of residuals testing (as opposed to waters) of WFS. Recent literature on toxicity testing includes considerable evidence of sediment bioassay analyses using not only the Microtox™ procedure but also a related proprietary option called Mutatox™, which intends to identify the presence or absence of potential contaminant mutagens as opposed to chemical toxins. Latter investigations appear to have similar goals, whereby semi-solid materials such as dredged residuals are being evaluated with regard to prospective disposal options and their prospective impacts (Bastian, 1996).

While higher life forms, including mammals such as rats, mice, etc., may commonly be used in conjunction with food and drug testing, fish are typically the most advanced organisms used with environmental tests. However, given the cost and complexity of using fish (e.g., fathead minnows, rainbow trout, etc.), the practice of bioassay testing has recently shifted towards less complex and less costly organisms, including invertebrates,

algae, fungi, and bacteria. Microbial bioassays have some decided advantages over animal and plant testing (Blaise, 1991). For instance, microorganisms are relatively easy to culture, and testing is generally less labor-intensive than when higher life forms are used. Due to their quick growth rate and small size, a statistically significant number of microorganisms can be economically used, negating the effects of individual variation. Microbial bioassays can be conducted rapidly, requiring a few minutes to a few hours as opposed to days, weeks, or even months for more complex organisms. Relatively small sample volumes are necessary. Finally, using microorganisms avoids the delicate and emotional issue of bioassay testing procedures, which might be linked to animal abuse concerns. The difficulty with microbial bioassays arises when one attempts to correlate the effects of environmental variables on such organisms with the possible effect on humans or other target species or environments. One way to get around this obstacle is to make conservative estimates of risk. To do this, microbial bioassays can be designed to be more sensitive to the particular environmental variable(s) than are humans or other target species (Bastian, 1996).

5.2.2 Microtox™ Bioassay

One bacterial bioassay which has been extensively used is the Microtox™ bioassay. The Microtox™ bioassay was the first commercially available acute, bacterial bioassay and is today the *de facto* standard with which other microbial bioassays are compared. It was first introduced in 1979 by Beckman Instruments (Bulich, 1979) and recently marketed by Microbics Corporation, Carlsbad, California. Since its introduction, Microtox™ protocols have been developed for samples of widely varying toxicity, ranging from contaminated soils to drinking water (Figure 3). The Microtox™ system utilizes the luminescent marine bacteria *Vibrio fischeri*. The enzymatic systems present in these organisms produce light when the appropriate environmental conditions are present (Nealson and Hastings, 1979; Ribo and Rogers, 1990). When the bacteria are negatively

High _____ Sample Toxicity Level _____ Low

Solid-Phase Test

(Microtox Acute Test)

- Soil
- Sediment

Basic Test

(Microtox Acute Test)

- Pure Compounds
- Septage
- WWTP Influent

90% Basic Test

(Microtox Acute Test)

- Process Waste Streams
- IPP Discharges
- Toxicity Baseline Studies

Inhibition Test

(Microtox Acute Test)

- TRE Testing
- Storm Water Runoff
- WWTP Effluent Screening
- Toxicity Source Testing

Comparison Test

(Microtox Acute Test)

- Final WWTP Effluent
- Final TRE Testing
- Final Storm Water Runoff
- Drinking Water

Chronic Test

(Microtox Chronic Test)

- WWTP Effluent
- Fate and Effect Studies
- Compliance Assurance
- Drinking Water

Figure 3 - Overview of Microtox™ Testing Protocols
(adapted from Bulich, 1979)

impacted, as by a toxin, the light they produce decreases. This decrease can be accurately measured, providing a quantitative determination of toxicity. Researchers have explored the biological effects of pollutants on this bacteria's ability to produce light for at least three decades (Serat, 1965). Simply put, the amount of light the bacteria produce is directly proportional to the bacterial activity. Using a control which provides the appropriate environmental conditions for the bacteria, light emission comparisons can be made between bacteria in the control and bacteria exposed to a sample. Negative impacts result in an inhibition of light production, and this decrease in emitted light intensity can be used to quantify toxicity in the sample (Salazar, 1997).

As the *Vibrio fischeri* organisms originated in a marine environment, the Microtox™ system was first applied to aqueous samples. To date, Microtox™ aqueous toxicity data has been gathered for over 1600 individual compounds (Kaiser and Palabrica, 1991). In addition, aqueous organic chemicals, municipal wastes, complex industrial wastes, and leachates from hazardous wastes have all been analyzed with Microtox™ tests (Munkittrick, 1991). More recently, contaminated sediments have been studied, both by measuring the toxicity of sediment extracts and by using a solid-phase test in which the organisms come into contact with the sediment directly.

Advantages of the Microtox™ test, include low cost, low time requirements, simplicity, and reproducibility. Potential weaknesses include a lack of sensitivity to certain compounds such as ammonia and cyanide, the necessary addition of 2% salt solution, pH constraints, and potential problems with samples that are colored or have significant turbidity, suspended solids, or hardness (Qureshi, 1982; Qureshi, 1984).

The Microtox™ test has also been compared with many other bioassay protocols, including those using bacterial, invertebrates, fish, amphibian, mammal, and plant species (Kaiser and Palabrica, 1991). Walker (1988) compared the relative sensitivity of 234 tests, using over 149 species, to phenol. It was seen that the Microtox™ assay was among the more sensitive species to the acute effects of phenol (Bastian, 1996).

Three bacterial groups, aerobic heterotrophs, *Nitrosomonas* and methanogens, were tested for their sensitivities to 50 to 100 chemicals including chlorinated aliphatic hydrocarbons, and halogenated and other substituted benzenes and phenols, and the results were compared to reported toxicity data for the Microtox™ test and fathead minnows. The Microtox™ test was shown to be as sensitive as *Nitrosomonas* and fathead minnows, and about one order of magnitude more sensitive than aerobic heterotrophs and methanogens. In an effort to streamline toxicity testing by establishing relationships between chemical toxicities to different organisms, correlations between the observed results were established with a 'good' correlation ($R^2=0.70$ to 0.82) found between Microtox™ and the other bacteria and the fathead minnow (Blum and Speece, 1991).

Samples of unknown composition were analyzed using the Microtox™ test and *Spirillum volutans* and, while the Microtox™ test was found to be more sensitive to several samples, a good agreement was found between the two tests (Coleman and Qureshi, 1985). However, Dutka and Kwan (1990) compared the response of the Microtox™ with *Spirillum volutans*, *Pseudomonas fluorescens*, and *Aeromonas hydrophila* species when exposed to samples of single metal concentrations as well as mixtures of these metals and determined that each bioassay had its own sensitivity pattern to toxicants (Salazar, 1997).

Microtox™ data were exhaustively reviewed by Munkittrick (1991), who found that for pure organics and complex effluents, the Microtox™ assay was generally at least as sensitive as rainbow trout, *Daphnia* species, and fathead minnows. Compounds which were exceptions to this trend included cyanide, chloroform, and phenol. For inorganics and organics including insecticides and herbicides, pharmaceutical wastes, and lipophilic contaminants, Microtox™ was generally less sensitive than the other species. Ammonia was singled out as having little effect on the Microtox™ organisms relative to the other species. It was noted that one study separated municipal wastes into soluble and insoluble constituents; Microtox™ was much more sensitive to the soluble constituents (Slattery, 1988).

Using toxicity data for 200 individual chemicals, a high collinearity has been demonstrated between Microtox™ toxicity and fathead minnow 96-hour lethality. Such relationships have also been demonstrated with Goldorfe fish and zebra fish, both of which have been used as standard test species in Europe. The least collinearity with Microtox™ toxicity has been seen with results from rat bioassays; this is not surprising, as rat bioassays have been shown not to correlate well with any other biological endpoint (Kaiser and Palabrica, 1991).

Samples of complex mixtures, including surface waters, municipal wastewaters, and industrial wastewaters and effluents have been tested with the Microtox™ assay (Bulich, 1984; Qureshi, 1984; Sanchez, 1988; Logue, 1989; and Levi, 1989). In general, the Microtox™ bioassay has a sensitivity level on the same order as other common bioassays. In one study, for example, eighty-two Brazilian industrial effluents were analyzed using Microtox™, *Spirillum volutans*, *Daphnia similis*, and several other bioassays (Sanchez, 1988). Microtox™, *Spirillum volutans*, and *Daphnia similis* results agreed well and allowed the researchers to rapidly screen for toxic discharges. Levi, constructed an on-line Microtox™ bioassay which provided the ability to monitor the toxicity of river water influent to a drinking water supply intake (Bastian, 1996).

Solids have also been evaluated using the Microtox™ test (Salazar, 1997). The traditional approach to testing the toxicity of solids has been the use of leachates or extractions, a method applied in the testing of a wide range of solids including different types of sludges, soils, sediments, and landfill leachates (Coleman and Qureshi, 1985; Kwan, 1990). The solution used for extracting pollutants from the solids plays a significant role in determining the effectiveness of the test to detect toxicity values. Solutions that have been tested include methanol, acetone, dimethyl sulfoxide (DMSO), dichloromethane and ethanol (Dutka and Kwan, 1988; Kwan, 1990; Schiewe, 1985). Applications reported include monitoring of a bioremediation site to assess cleanup efficiency (April, 1990) and testing the treatment of a hazardous waste through land application by monitoring soil-water toxicity levels (Casarini, 1991). Another approach

at evaluating solid toxicity has been recently developed using the Microtox™ test in which the bacteria are allowed to have direct contact with the solids for a period of time, and then are separated from the solids by filtration and analyzed for light production (Tung, 1990).

In short, the sensitivity of the Microtox™ bioassay is comparable to that of most other commonly used species, and is great enough to be of value in most toxicity testing scenarios (Bastian, 1996). The optimal strategy, however, is the use of more than one bioassay species, even including species of widely differing biological complexity, in a 'battery of tests' approach (Dutka and Kwan, 1988). In this way, the different tests can be used in a complementary fashion. Pastorok and Becker (1990) reached a similar conclusion when comparing organically-extracted sediments from Superfund sites in Puget Sound using Microtox™ and six other sediment toxicity bioassays.

5.2.3 Microtox™ Bioassay Testing of Foundry Wastes

Limited research has been done using the Microtox™ test to assess the toxicity of WFS. One study performed by Pennsylvania State University (Westervelt, 1988) compared the toxicity evaluations of Microtox™ (using *Vibrio fischeri* bacteria) with *Daphnia Magna*. Samples from 33 Pennsylvania foundries were collected and composited into four categories: sand wastes from all of the foundries, sand wastes from only non-ferrous foundries, baghouse dust from all foundries, and sludge from all foundries. These composite samples were mixed in quantities proportional to the output of the several foundries; the composite sand, for example, consisted of 95% ferrous foundry waste and 5% non-ferrous foundry waste. Unfortunately, wastes from ferrous foundries were not isolated for testing, nor was it noted that only greensand process wastes were used. Each of the four sample types were tested for bulk and leachate chemical concentrations and for leachate toxicity. Different leachate types were used: saturated paste leachates, 5:1 and 20:1 water-solid ratio leachates, and TCLP leachates. Bulk analysis revealed that silica comprised over 95% of the sand wastes and approximately 75% of baghouse dust and sludge wastes. Additional major cations included aluminum, calcium, iron,

potassium, magnesium, sodium, and zinc. Lead was also found at a concentration of approximately 3.5% in the sludge samples. Other than lead in the sludge samples, only trace levels of the metals included in primary drinking water standards (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver) were detected. Sludge samples leached the greatest amount of aluminum, cadmium, nickel, lead, sulfate, and chloride. Baghouse dust leached the greatest amount of iron. Manganese and zinc were highest in non-ferrous foundry sand leachate. The composite sands leached the fewest metals (Bastian, 1996).

Microtox™ toxicity results are listed in Table 8. The unit used is toxic units (TU), defined as the reciprocal of the dilution that causes 50% inactivation of the test population. It was seen that after 5 minutes, the composite sand samples exhibited the greatest Microtox™ toxicity for each leachate. That toxicity levels remained relatively constant over the 30-minute time period, suggests the source of toxicity was organic, as test organisms typically display effects produced by organic compounds within five minutes. After 15 and 30 minutes, the greatest Microtox™ toxicity by far was displayed by the non-ferrous sands. An increase in toxicity after five minutes suggests metal contamination, as metal-related inhibition often takes longer than 5 minutes to develop (Bastian, 1996). *Daphnia magna* acute toxicity was generally limited to the non-ferrous sand samples. *Daphnia magna* results indicated these organisms were probably sensitive to metals, as inhibition was observed with the non-ferrous sands, but were not sensitivity to the organics detected by the Microtox™ test in the ferrous sands. Literature toxicity data for *Daphnia* species and Microtox™ organisms were compared with leachate metal concentrations. Zinc and, to a lesser extent, cadmium and copper were identified as potential toxins to *Daphnia* and Microtox™ bacteria in non-ferrous sand leachate samples. No other leachate samples contained metals at levels which suggested significant metal toxicity (Salazar, 1997).

**Table 8: Microtox™ Toxicity of Foundry Wastes
(after Westervelt, 1988)**

5-minute results (TU)			
Leachate Source	Saturated Paste	5:1 water:solid	20:1 water:solid
Composite sand	12.5	7.4	2.7
Non-ferrous sand	8.3	2.9	1.6
Baghouse dust	2.1	0.7	0.4
Sludge	1.7	0.6	0.4
15-minute results (TU)			
Leachate Source	Saturated Paste	5:1 water:solid	20:1 water:solid
Composite sand	11.7	6.9	2.1
Non-ferrous sand	53.3	7.7	3.9
Baghouse dust	2.5	0.8	0.4
Sludge	1.5	1	0.6
30-minute results (TU)			
Leachate Source	Saturated Paste	5:1 water:solid	20:1 water:solid
Composite sand	12.6	7.6	2.1
Non-ferrous	263.7	18.3	--
Baghouse dust	3.4	1	--
Sludge	1.6	2	0.7

The hypothesis concerning metal contamination was tested by adding EDTA, a compound which chelates (captures) metals thus preventing them from causing toxicity, to foundry waste samples. As shown by the Microtox™ data in Table 9, composite sand toxicity increased slightly upon addition of EDTA, while non-ferrous sands to which

**Table 9: Microtox™ Toxicity of Foundry Wastes Upon Addition of EDTA
(after Westervelt, 1988)**

Leachate Source	Toxic Units (TU)		
	5-minute	15-minute	30-minute
Composite Sand	12.8	11.5	11.9
Composite Sand with EDTA	17.2	14.3	14.7
Non-ferrous Sand	3.2	8.1	17.9
Non-ferrous Sand with EDTA	0.89	1.0	1.1

EDTA had been added were less toxic than the unaltered sands after 5 minutes and remained at that low level throughout the 30 minute test. These findings confirmed the indications of toxicity source: organics in the composite sand samples and metals in the non-ferrous sand samples (Bastian, 1996).

Due to the large ratio of ferrous sands to non-ferrous sands, it is possible that the Pennsylvania composite sand samples had low enough heavy metal concentrations that only toxicity from organics was exhibited by the Microtox™ system. When only non-ferrous sands were tested, however, high Microtox™ toxicities likely resulted from metals rather than organic binders or additives. As *Daphnia magna* toxicity was only observed in non-ferrous sand leachate samples, it is possible that these organisms were not sensitive to the organics present in the composite sands but were affected by the metal-associated toxicity of the non-ferrous sands. The reasons for the lower Microtox™ toxicity of the non-ferrous sands in the 5-minute test, with the addition of EDTA, were not clear (Bastian, 1996).

The organics utilized in casting systems are associated with greensand binder additives (sea coal, starch, pitch, asphalt, and petroleum distillates) and with core binders. Such additives would be found in ferrous and non-ferrous systems; the amount of organics remaining on the sand would depend on melting temperature, binder type, casting size and shape, and a multitude of other site-specific variables. Toxic metals would probably only be found in appreciable quantities in non-ferrous foundries: in particular, brass contains copper, zinc, and lead; bronze is comprised primarily of copper and tin (Bastian, 1996).

Bastian (1996) evaluated the ability of the Microtox™ bioassay to assess the toxicity of WFS intended for beneficial reuse as construction materials. WFS from ferrous and non-ferrous foundries were tested to determine which sands would be suitable for reuse by comparing Microtox™ responses to responses from virgin sands commonly used in foundry and construction applications. Results indicated the binder type used in each process was a determining factor in the measured inhibitory effect of these sands, with

certain processes resulting in higher concentrations of organic residuals which in turn resulted in a more toxic response. WFS from aluminum and steel foundries were also tested and determined to cause significant bacterial inhibition, but results were based on a limited sample set. The study effectively showed the ability of the Microtox™ test to effectively ‘fingerprint’ the ‘toxicity’ of foundry residuals as compared to virgin sand materials (Salazar, 1997).

5.2.3.1 Sensitivity of the Microtox™ Bioassay to Chemicals Typically Found in WFS'

Leachates

Acute toxicity data for the Microtox™ test have been compiled for a vast array of compounds (Salazar, 1997). The bacterial sensitivity to these compounds allows for a detectable inhibitory effect when exposed to very low concentrations, with minimum detectable concentrations determined by a 2% light inhibition. Most toxicity data, however, have been historically reported as EC₅₀ values, the concentration of a compound causing a 50% reduction of the light emitted by the bacteria over a given exposure time. For suspected contaminant concentrations greater than the EC₅₀, testing of samples at different levels of dilution can yield valuable information on the approximate concentration values present in the sample. For samples containing compound concentrations much lower than the EC₅₀, however, the minimum detectable concentration values would be much more appropriate for determining the applicability of the test. These values are not available in the literature for most compounds, so an attempt to use the EC₅₀ values as indicators of sensitivity to lower concentrations must be made (Salazar, 1997). The ratios between EC₅₀ values and minimum detectable concentrations vary greatly between compounds, making it difficult to predict with accuracy the minimum detectable concentration based on the EC₅₀ values. For example, the Microtox™ minimum detectable concentration of toluene is 8 mg/l, about one half of the EC₅₀ value. For acetone, on the other hand, the Microtox™ minimum detectable concentration is 1240 mg/l, one tenth of the EC₅₀ value (Bulich, 1986).

Table 10 (Salazar, 1997) lists available lowest reported Microtox™ EC₅₀ values (Bastian, 1996; Bitton and Koopman, 1986; Blum and Speece, 1991; Boane, 1997; Dutka and Kwan, 1982; and Kaiser and Palabrica, 1991) for contaminants detected in WFS, as well as the maximum reported concentrations in the literature from leachate analyses (Blaha, 1986; Ham, 1993a; Javed, 1993). Lysimeter data was used for the maximum concentrations of TOC and COD. Fluoride and sulfate maximum concentrations from lysimeter analysis exceeded leachate concentrations, with values of 120 mg/l and 1220 mg/l, respectively (Boyle and Ham, 1979). Also listed are the regulatory levels for these compounds, as listed in different sources of criteria to be used in the evaluation of these wastes (Salazar, 1996; Blaha, 1986; 40 CFR 261; 40 CFR 141; 40 CFR 143; Ham, 1993a; 329 IAC 2). All listed EC₅₀ values are for either 5- or 15-minute exposures.

Data from the analysis of WFS used in construction of an embankment on CR 206 near Auburn, Indiana (described in detail in Chapter 6) for constituents (provided as confidential data) as required by the Indiana waste characterization criteria, indicated maximum concentrations similar to the detected maximum concentrations listed in Table 10. Values detected at higher concentrations than those listed in Table 10 were still lower than the given regulatory levels. This similarity in detected concentrations granted the use of reported values as a guide in evaluating the performance of the WFS used in the CR 206 demonstration project (Salazar, 1997).

Table 10: Contaminants Detected in WFS: Regulatory Levels, Lowest Reported EC₅₀ Values, and Maximum Concentrations Detected in Leachate Analyses (Salazar, 1997).

Contaminant	Regulatory Level (mg/l)	Lowest Reported Microtox™ EC ₅₀ Conc. (mg/l)	Max. Conc. Detected (mg/l)
Metals			
Arsenic	0.05 ¹	N/R	0.02
Barium	2.0 ²	N/R	0.5
Boron	1.0 ¹	N/R	0.4
Cadmium	0.005 ²	10	0.05
Chromium	0.1 ²	13	0.04
Copper	1.0 ²	3.8	0.12
Iron	0.3 ²	N/R	71
Lead	0.05 ¹	30.1	0.09
Manganese	0.05 ²	N/R	6.5
Mercury	0.002 ²	0.03	0.002
Nickel	≤0.2 ⁷	23	<0.1
Selenium	0.05 ²	N/R	0.03
Silver	0.1 ²	0.86	<0.05
Zinc	5.0 ²	1.4	60.0
Other inorganics			
Chloride	250 ²	N/R	<100.0
Cyanide	0.2 ²	2.8	0.08
Fluoride	2.0 ²	N/R	16
Sodium	≤250 ⁷	N/R	35.98
Sulfates	250 ²	N/R	85.03
Sulfide	≤1 ⁷	N/R	<1.0
TDS	≤500 ⁷	N/R	236
Organics			
1,1,1-Trichloroethane	0.2 ²	8.04	0.049*
2,4-Dimethylphenol	N/A	2.44	0.12*
2-Methylnaphthalene	N/A	N/R	0.32*
Acetone	0.25 ⁵	12,400	<0.1*
Benzene	0.005 ²	2	11
Benzoic acid	N/A	16.9	N.D.*
COD	100 ⁶	N/R	290
Dimethylphthalate	N/A	16.15	0.061*
Ethylbenzene	0.7 ²	6.55	0.0006*
o-Cresol	200.0 ³	15.4	0.11*
p-Cresol	200.0 ³	1.3	0.045* (m/p)
m-Cresol	200.0 ³	6.82	
Naphthalene	N/A	0.81	0.48*
Phenanthrene	N/A	0.04	<0.03*
Phenol	14.4 ⁴	18	0.34*
Tetrachloroethene	0.1 ⁴	13.4	0.007*
TOC	N/A	N/R	120
Toluene	1.0 ¹	17.1	0.0047*
o-Xylene	10.0 ² (total)	9.25	0.0017*
m/p Xylene		5.7	0.0023*
Parameter	Acceptable Range	Desirable Range	Range Measured
pH	6.5-8.5 ²	6.0-8.0	7.8-9.8
N/A – Not Available: No regulatory level available for this contaminant in any of the referenced criterion.			
N/R – None Reported: No EC ₅₀ value reported for this contaminant.			
1: Drinking Water Standard as of 1989 (Blaha, 1996).			
2: Drinking Water Standard as of 1996 (40 CFR 141, 40 CFR 143).			
3: TCLP Criteria (40 CFR 261)			
4: TCLP Criteria (Ham, 1993)			
5: Trigger Level (Ham, 1993)			
6: Common concentration found in groundwater, given only as a comparison value.			
7: Indiana Waste characterization: Type IV Criteria (329 IAC 2)			
*Maximum values detected in phenolic urethane sand were used instead of other (sometimes higher) reported maximum concentrations			

CHAPTER 6: CR 206 FIELD DEMONSTRATION PROJECT

6.1 Introduction

With laboratory evaluations complete, indicating that WFS from ferrous foundries could provide the necessary engineering properties for a highway embankment (Javed, 1993; Javed and Lovell, 1994; Javed, 1994) and that the complementary (to required regulatory testing) Microtox™ test could screen the toxicity of the WFS to prevent a negative environmental impact on the site (Bastian, 1996), the next step was the construction of a full scale embankment using WFS, with geotechnical and environmental instrumentation installed to evaluate its performance. During July and August of 1996, approximately 42,800 m³ of WFS from the Auburn Foundry Inc. monofill (single waste landfill) in Auburn, Indiana was placed as structural fill in a 105 m long embankment section on County Route (CR) 206 in DeKalb County, Indiana (Figure 4). CR 206 is a two-lane rural road which was built for access to Steel Dynamics, Inc. (SDI), a steel recycling plant. Most of the steel coming into the plant is hauled in heavy tractor trailers.

Additionally, two control embankment sections were constructed for comparison with the WFS embankment section. The southernmost section was built using a clay soil from a local borrow pit and is representative of typical fill material used by INDOT. Adjacent and north of the WFS embankment, an embankment section was constructed using natural (virgin) sand which meets INDOT specifications for 'B-borrow' sand. B-borrow sand is often used as a structural fill material behind structures which require freely-draining backfill, such as retaining walls and bridge abutments. B-borrow was used on this project as it was thought that the grain-size distribution of the B-borrow sand would more closely approximate the distribution of WFS.

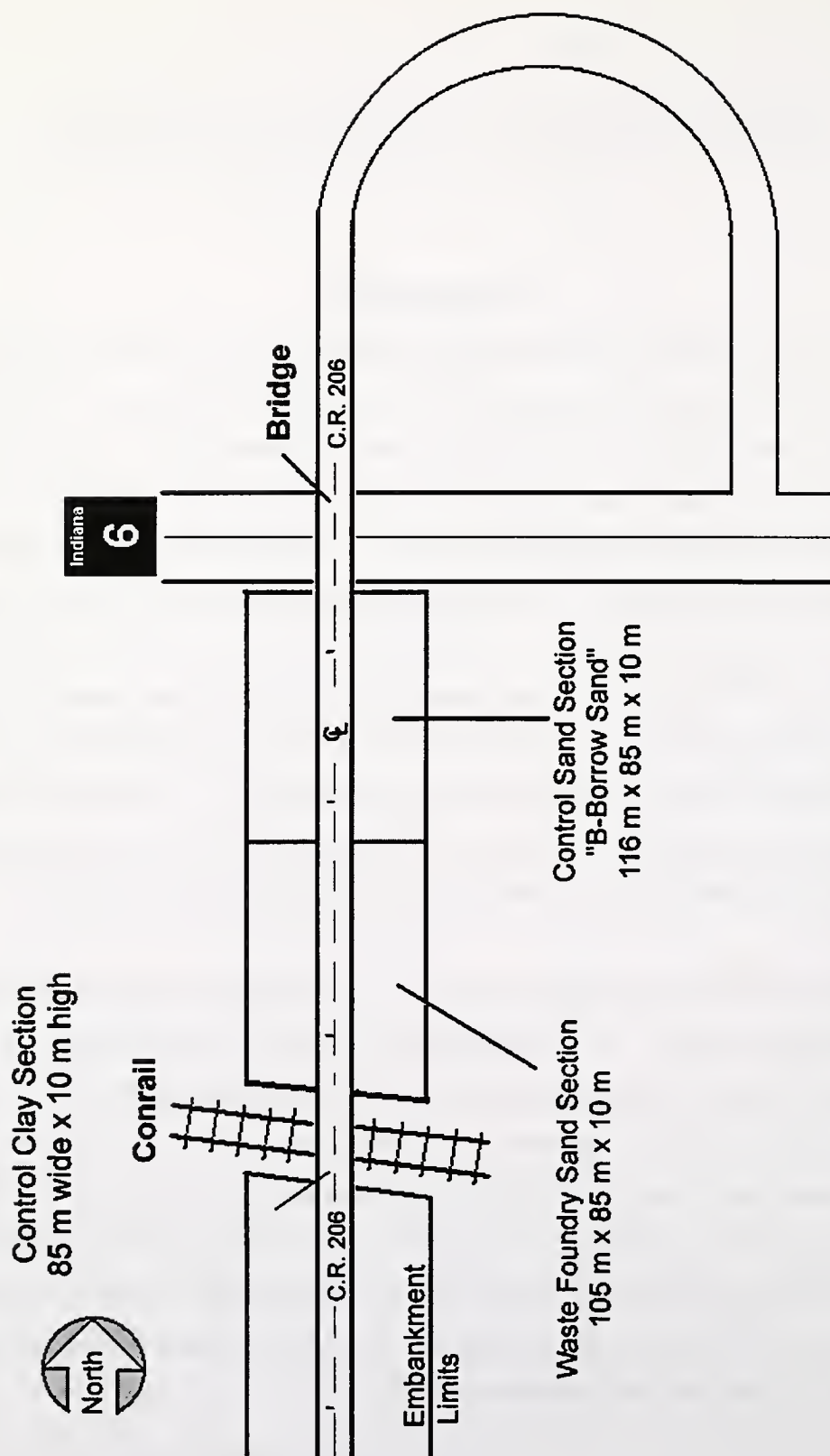


Figure 4 – CR 206 Project Section Layout

The project site appears on the USGS topographic map (Butler West quadrangle map, Township 34 North, Range 14 East, Section 10) as very flat, with no major drainage routes visible. Observations made in the field before and during construction indicated that drainage of the land is accomplished mainly by farm field tiles and a ditch running parallel to the railroad tracks. A number of wetland areas are designated on the topographic maps, some of which were moved as part of the project construction. The poor drainage and wetlands in the project area suggested there could be large sections of soft organic peat layers which could cause construction problems. INDOT soil borings directly below the proposed roadway, however, did not indicate peat deposits although peat bogs were found in other areas of the project site.

As noted in Chapter 1, the WFS used for this project were classified as IDEM Type III and IV special wastes and were obtained from the Auburn Foundry monofill. Auburn Foundry, Inc. is a large gray iron foundry which used a phenolic urethane compound as the principal core binder at the time the waste sand was disposed of. The phenolic urethane binder, usually known as an Ashland or Isocure process, belongs to the 'cold box organic binder' group. It includes a combination of phenol formaldehyde resin and polymeric isocyanate. The catalyst is vaporized before application to produce a rapid cure, and usually utilized catalysts are gaseous TEA (triethylamine) or DMEA (dimethylethylamine) (Bastian, 1996; Clegg, 1991). Castings at this foundry ranged between 0.5 and 32 kg in weight, and totaled approximately 907 Mg of metal cast per day. The sand was obtained from the foundry-owned monofill, where approximately 113 to 136 Mg of additional WFS per day were stockpiled as they came out of the plant. The Auburn monofill is located 27 km from the project site and is not covered, allowing weathering of the sand by exposure to rain and other weathering factors.

The purpose of the field demonstration project was to evaluate the geotechnical and environmental impact of using WFS as an embankment construction material. While the purpose of this report is to primarily discuss the environmental characteristics and impact of the WFS on the site, the geotechnical work plan and geotechnical performance results

are briefly mentioned. These results are included as the decision to use WFS requires a holistic approach, i.e. WFS not only should not pose a negative environmental impact on a site, but must also provide engineering properties comparable to presently used construction materials. Other concerns regarding the reuse of WFS include regulatory and liability issues, quality control issues, and economic considerations. As noted in the literature review in Chapters 3 and 5, few embankments have been constructed using WFS, therefore, for this demonstration project both geotechnical and environmental performance were evaluated. For a detailed report on the geotechnical performance of the embankment materials used in this demonstration project see the companion final report (Fox and Mast, 1998). This demonstration project was a joint effort between INDOT, INCMA and Purdue University.

6.2 Geotechnical Work Plan

As noted in the introduction to this chapter, the main embankment, located between US 6 and the nearby rail line, was built in two sections, one with WFS and the other with natural (B-borrow) sand. A second embankment was constructed on the south side of the rail bridge using clay borrow. The B-borrow and clay fills served as control sections with which to compare the performance of the WFS section. Each embankment is 10 m high at the centerline, 85 m wide at the base, and has 3H:1V side slopes.

A geotechnical investigation of the project site was conducted in August 1995. The soil borings and Standard Penetration Test results revealed the following soil profile under the main embankment: medium stiff loam (depth=0.3 to 0.6m), loose sand (0.6 to 1.5m), stiff loam (1.5 to 2.4 m), medium dense sand (2.4 to 3.7 m), hard clay (3.7 to 8.2 m), and medium dense sand and gravel (8.2 to 10.7 m). The groundwater table lies at a depth of approximately 1.5 m. One of the concerns during the investigation was the possible existence of organic soils and peats in the subsurface, however the borings directly below the proposed roadway did not indicate any such deposits. The soils in this part of Indiana are primarily glacial in origin.

In June 1996, a field test pad was constructed at the Auburn monofill to develop a method specification for compaction of WFS in the field. A smooth drum vibratory roller and a heavy (36,000 kg) rubber-tire roller were used for comparison. It was determined that the WFS should be placed in 0.2 m lifts at a water content between 12 and 15%, and compacted using 6 passes of the rubber-tire roller. The natural sand and clay sections of the embankments were compacted using the vibratory roller and a sheepsfoot roller, respectively.

A variety of instrumentation was used to monitor the geotechnical performance of the embankment (Figure 5). Prior to construction, two piezometers and two vertical inclinometers were installed in the subsurface. The first truckload of WFS arrived on site 15 July 1996 and was placed directly on the natural subgrade. No liner materials (e.g., compacted clay, geomembrane) were installed underneath the WFS. The geotechnical instrumentation plan called for four nests of settlement plates, two in the WFS and one in both the natural sand and clay sections. For each nest, plates were placed at the bottom, middle, and top of the embankment. Horizontal inclinometers were also installed at the bottom and near the top of the WFS section to provide redundant measurements of settlement. Total pressure cells were placed underneath the WFS section to measure the stress applied to the foundation. While the contractor completed the bridges, a sealed double-ring infiltrometer (SDRI) was installed to measure the in situ hydraulic conductivity of the compacted WFS and infiltration was monitored over a 5-week period. In addition, unit weight measurements were taken during construction to compare laboratory Proctor compaction curves and to evaluate the reliability of different measurement instruments (sand cone, nuclear density gage, and Speedy Moisture Meter) for WFS. Placement of the WFS was completed on 15 August 1996. A 0.6 m thick layer of top soil was placed over the WFS and natural sand sections to reduce erosion and support vegetation. The pavement was laid and CR 206 was opened to traffic in late November, 1996. In February 1997, three borings were drilled through the embankment to evaluate the insitu properties of the compacted WFS, natural sand, and clay borrow using the standard penetration test (SPT). Project monitoring continued through June 1998.

C.R. 206 Foundry Sand Project

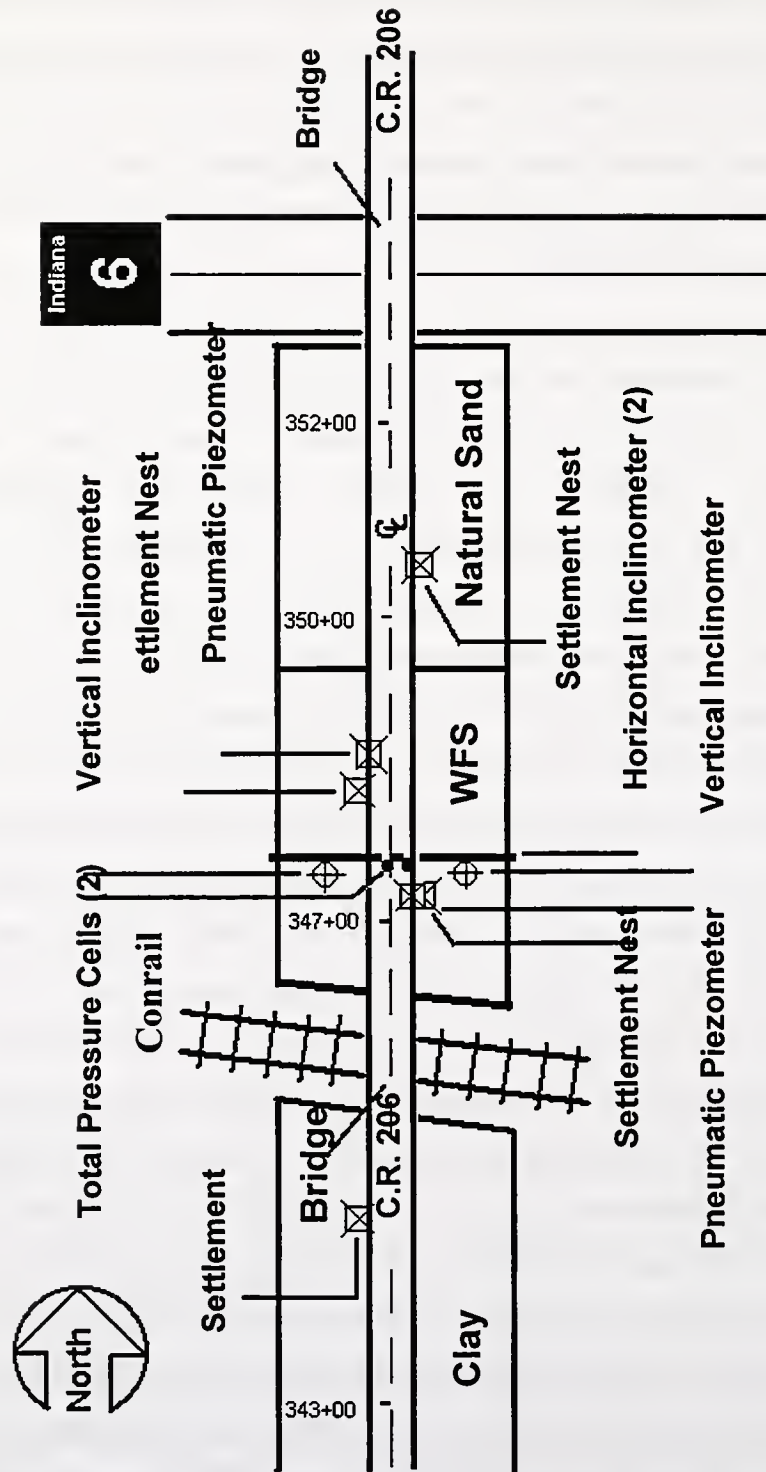


Figure 5 - Geotechnical Instrumentation

6.3 Environmental Work Plan

To establish background water quality at the project site, water samples were collected from six groundwater monitoring wells (Figure 6) which were installed prior to embankment construction. Four wells (WF1, WF2, EF1, and EF2) were installed adjacent to the WFS embankment and the other two wells (WV and EV) were installed adjacent to the natural sand embankment. Local groundwater flows from west to east, making the west wells up-gradient from the embankment and allowing for assessment of groundwater quality before and after exposure to the WFS. In addition, two lysimeters were installed in the WFS (FLY) and natural sand (VLY) sections of the embankment. The lysimeters consisted of 20.3 cm diameter corrugated slotted PVC pipe placed at a 0.01 slope 15.2 m into the embankment and perpendicular to the centerline of the road. The pipe rested on a 40 mil HDPE geomembrane to maximize leachate collection (Figure 7). The two lysimeters were identical, except that the natural sand lysimeter was wrapped in a fabric pipe sock to prevent clogging of the slots by fines contained in the sand.

Pre-construction groundwater sampling (9 July 1996) was limited to four samples due to difficulties encountered during installation of the wells as a result of heavy rains immediately prior to construction. This lack of data was not detrimental to the study given that up-gradient groundwater samples were collected throughout the course of the project providing information on background water quality. Groundwater samples were collected weekly during and after construction from 15 July to 29 October 1996. Thereafter, monthly sampling was considered adequate to characterize site water quality.

Natural sand lysimeter leachate collections began 5 September 1996 and continued at the same frequency as the groundwater sampling. Leachate collections from the WFS lysimeter could not begin until 2 October 1996 due to a temporary soil cover over the sealed lysimeter outlet. After 2 October 1996, WFS lysimeter collections proceeded at the same frequency as groundwater sampling.

C.R. 206 Foundry Sand Project

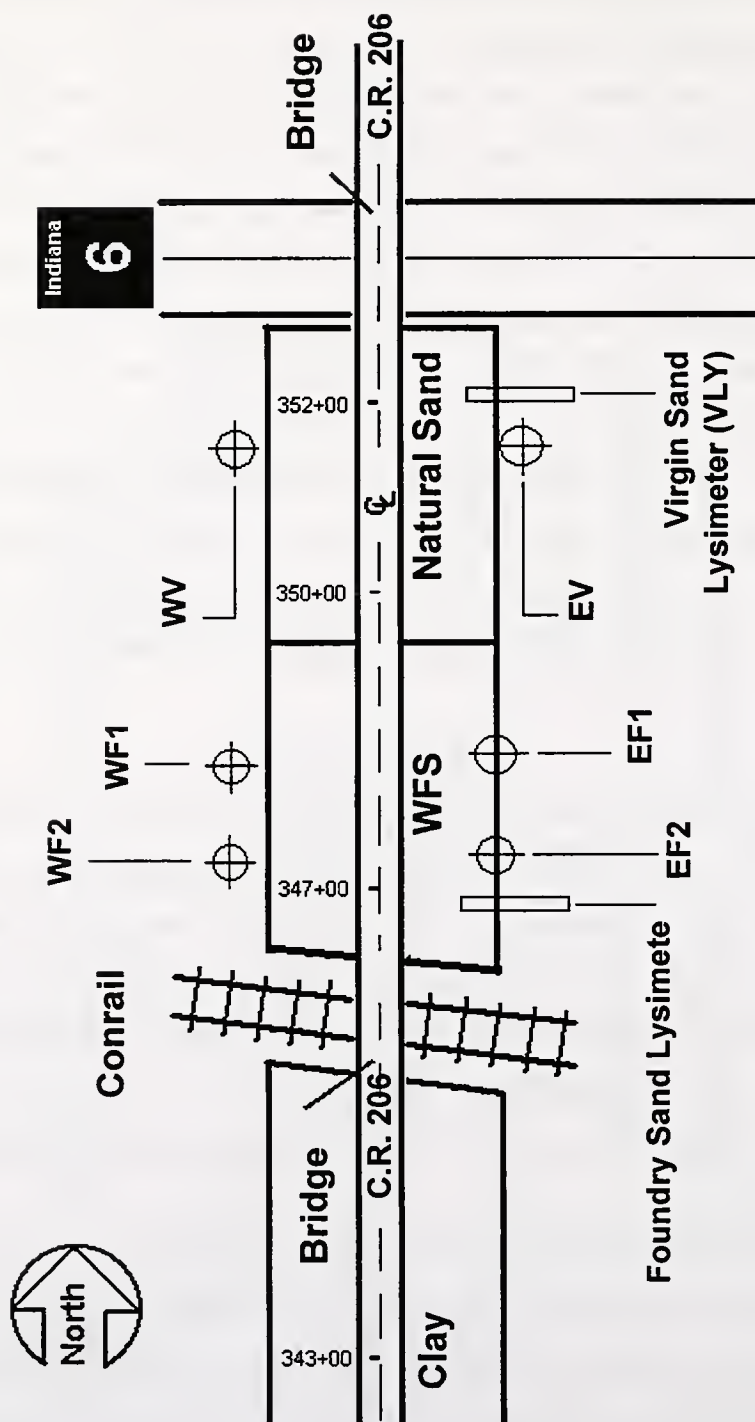


Figure 6 - Groundwater Monitoring

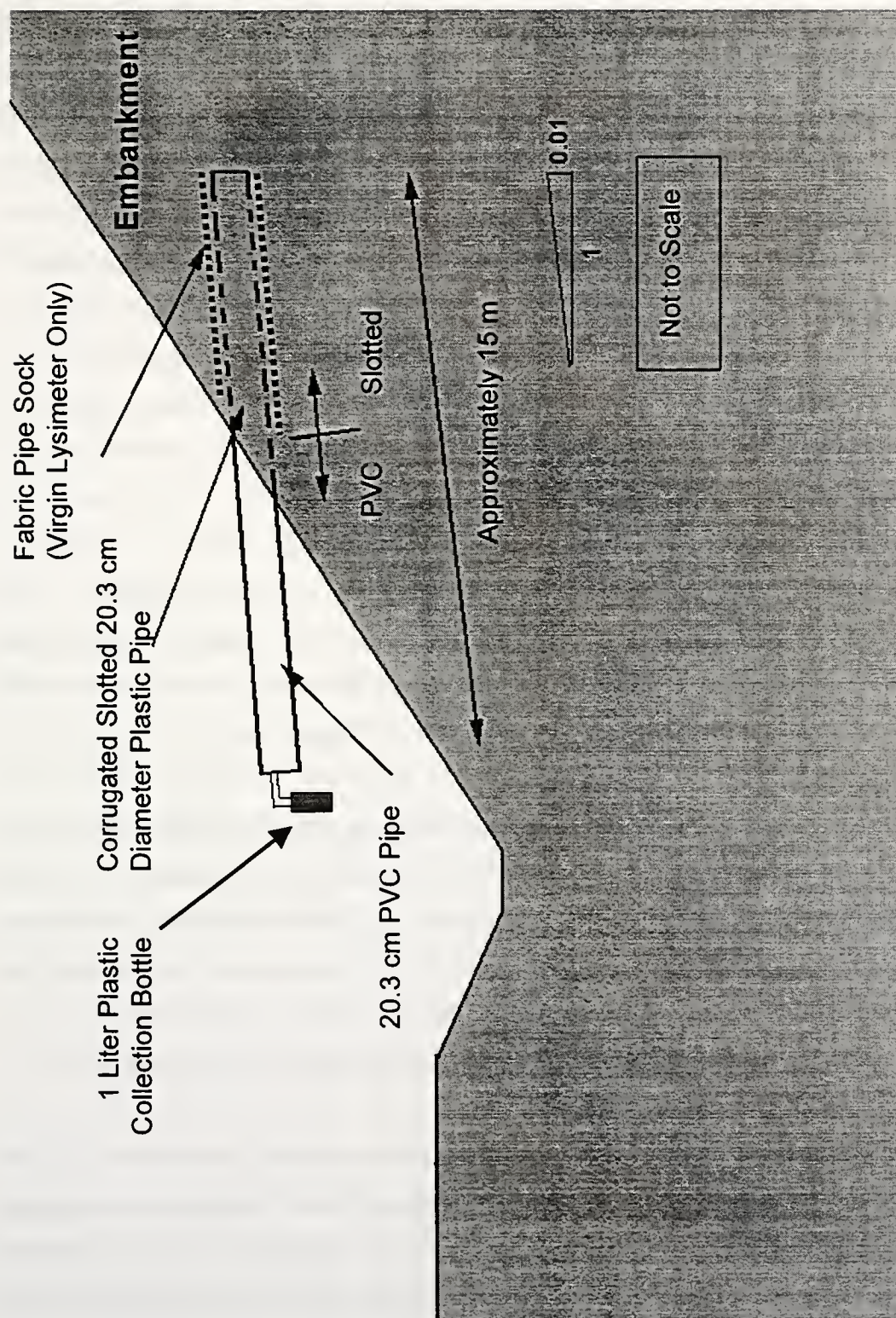


Figure 7 – Lysimeter Layout: Cross-Section
(after Salazar, 1997)

In addition to the collection of groundwater and lysimeter leachate samples, 17 WFS samples were collected for leachate testing from the Auburn monofill prior to construction (13 July 1994 to 16 March 1995) to characterize the stockpile. Furthermore, 31 WFS 'grab samples' were collected twice daily at the job site during construction. Leachates from the WFS samples were generated following a protocol similar to the EP water leachate test (329 IAC 10-7-4) in an attempt to simulate field conditions. The Auburn monofill was sampled at random from the surface to a depth of 0.6 m, while job site samples consisted of WFS from the entire 10.7 m depth of the monofill.

Testing of groundwater and lysimeter leachate samples, as well as leachates generated from WFS job site samples, consisted of the Microtox™ 90% Comparison Test (suggested for low toxicity samples) and Nitrotox bioassays. Additionally, ion chromatography (IC) testing was performed for select ions (F^- , NO_3^- -N, Cl^- , and SO_4^{2-}) using a Dionex GP40 Ion Chromatograph and inductively coupled plasma (ICP) spectrophotometer testing using a Thermo Jarrel – Ash ICP, to test for various metals (Ag, As, Ba, Cd, Cr, Cu, Mn, Ni, Pb, Se, and Zn). WFS leachates from the weathered stockpile samples were tested using the same Microtox™ procedure.

The prototype Nitrotox bioassay, which uses nitrifying organisms (99% *Nitrosomonas* bacteria) to evaluate toxicity, was included as a comparison bioassay test to the established Microtox™ test. Past research (Bastian, 1996) has indicated the sensitivity of the Nitrotox test is at least comparable to, and perhaps greater than, that of the Microtox™ bioassay. Side-by-side testing of the two bioassays allowed for comparison of their respective sensitivities to chemicals found in WFS from a ferrous foundry.

Selection of ions and metals for testing was based on contaminants detected in WFS (Javed, 1993; Salazar, 1997) and which are common, based on regulatory compliance data, to ferrous foundries. The ions were used as 'tracers' to track migration of contaminants (e.g. organics) from the WFS embankment into the lysimeter leachate and

- groundwater samples. While testing for specific organics was not part of this project, if ion migration could be shown and an increase in inhibition was displayed by the Microtox™ test (after 5-minute exposure) in the down-gradient wells and WFS lysimeter, it would be likely that organics were leaching from the WFS. Prior research (Westervelt, 1988) of toxicity evaluations using Microtox™ with *Daphnia Magna* have indicated that toxicity values detected in WFS (95% ferrous) were probably due to organic content, as maximum detected inhibition was measured within the 5-minute exposure. An increase in toxicity after five minutes suggests metal contamination, as metals often take longer than five minutes to exert their effects (Bastian, 1996).

6.3.1 Methods and Materials

6.3.1.1 Introduction

This section addresses the sample types collected (previously noted in this Chapter) and collection techniques; procedures for leachate generation; and protocols for the Microtox™ and Nitrotox bioassays, ion chromatography (IC) testing and inductively coupled plasma (ICP) spectrophotometer testing. Three types of samples were collected during this study: 1) WFS samples collected before and during construction; 2) groundwater samples collected before, during, and after construction; and 3) lysimeter samples, collected during and after construction.

Pre-construction WFS stockpile samples were random samples, one to five pounds each, collected from the Auburn monofill over a 9 month period. This monofill was the source of WFS used in the CR 206 demonstration project. During construction, WFS job site samples were collected from the haul trucks as the WFS were unloaded at the site. One sample was collected each morning and afternoon as the WFS were delivered. Samples were collected in glass pint-size canning jars. The daily samples were stored at the site and transported to Purdue University once a week, where they were kept at 4°C until

tested. All WFS samples were labeled with the sample origin, the date, and the time of collection (Salazar, 1997).

Groundwater samples were collected using a PVC collector (approx. 0.18 liters) attached to a metal chain. Five collections were performed at each well, the first three of which were used to rinse the collector and discarded to avoid contamination from the previous sample. The other two collections, approximately 0.36 liters, were then poured into glass canning jars, labeled by date and sample location, and brought to Purdue University to be stored at 4°C until tested (Salazar, 1997).

As previously noted, two lysimeters were installed at the site. One lysimeter (FLY) was installed in the WFS embankment and the other lysimeter (VLY) was installed in the natural sand embankment. To ensure that the leachate would not simply run off the end of the membrane without going into the slotted pipe, a metal clamp was placed at the point where the slotted pipe joined the PVC pipe (Figure 7) making a seal at the edge of the geomembrane. This created a cavity in which the leachate could be collected and transferred from the slotted pipe into the PVC outlet pipe. As the amount of liquid coming through the lysimeters was limited, collection bottles were installed at the end of the outlet pipes. A small hole was made in each cap, and a PVC elbow connector was glued and attached to a 1 liter plastic bottle. To avoid liquid buildup inside the lysimeter, one small hole was put near the top of each bottle to allow for pressure release and overflow of the collectors. Lysimeter leachate samples were transferred directly from the collection bottles into glass canning jars, labeled by date and sample location and brought to Purdue University to be stored at 4°C until tested (Salazar, 1997).

6.3.1.2 Bioassay Leachate Generation

The WFS leachates for bioassay testing were generated following a protocol similar to the EP water leachate test, in an attempt to simulate field conditions. A 1:4 solid-liquid ratio was used by placing 20.00 ± 0.05 grams of WFS and 80 ± 1 ml water in a flask. The 1:4 solid-liquid ratio was recommended by Microbics Inc. when performing the

Microtox™ acute test (used in this project) and is considered conservative. This ratio has also been used for leachate generation in the Serial Batch Leach Test (SBLT) to test the success of Solidification/Stabilization (S/S) for treating explosives contaminated soils (Cullinane and Channel, 1996).

For the leachate generated for the Microtox™ test, a saline solution (2% NaCl) was added to the water which provided osmotic protection for the luminescent bacteria. Leachates to be used for ion chromatography were performed with nanopure water. Each flask was covered in parafilm, manually agitated to break down large clumps of sand, and then placed on a shaker table at 175-200 RPM from 18±2 hours. No mechanical breaking or crushing of the sand was performed to represent actual site conditions. After shaking, the samples were allowed to settle and 30 to 40 ml of the supernatant was poured into polycarbonated centrifuge tubes and centrifuged for 16 minutes at 10,000 RPM. The centrifuge supernatant was then filtered using a 1.5 µm pore size glass fiber filter and a 0.45 µm membrane filter to remove fines. The pH was measured and recorded. Although for optimum sensitivity, the desired pH for the Microtox™ test is between 6.5 and 8.0 (Azur Environmental, 1997) (between 7.5 and 8.0 for the Nitrotox test), no pH adjustments for the Microtox™ samples were performed as previous studies have shown little effect on measured toxicity levels due to pH differences (Coleman and Qureshi, 1985). Samples were transferred to borosilicate glass vials, covered with parafilm, and capped. For the bioassays, the samples were either tested immediately or stored at 4°C for no more than 72 hours prior to testing. Samples to be analyzed by ion chromatography and inductively coupled plasma spectrophotometer testing were stored at 4°C until tested (Salazar, 1997).

All groundwater and lysimeter leachate samples were filtered using a 1.5 µm pore size glass fiber filter. The pH of each sample was measured and recorded. The samples were then transferred into borosilicate glass vials, covered in parafilm, and capped. For the Microtox™ and Nitrotox tests, samples were either tested immediately or stored at 4°C for no more than 72 hours prior to testing (Salazar, 1997).

6.3.1.3 Microtox™ Testing Protocol

Based on the low expected sample toxicity levels, the 90% Comparison Test (or Microtox Acute Test) was used to test WFS leachates and groundwater and lysimeter leachate samples (Salazar, 1997). This test is recommended for final wastewater treatment plant effluents, final stormwater runoff, drinking water, and other low toxicity samples. The test is a comparison between five replicates of a control, a 2% saline solution in which the bacteria will live with no negative inhibitions, and five replicates of the sample. The testing protocol can be described as follows (Azur Environmental, 1997). The Microtox™ reagent (*Vibrio fischeri*) was supplied freeze dried and was reconstituted using 1 ml of reconstitution solution. This and all other solutions were maintained at 15°C throughout the test. The reconstituted reagent was diluted and 100 µl of it distributed into each of ten cuvettes. After 15 minutes of temperature equilibration, light emission from the cuvettes was measured. 0.9 ml of the sample was then added to each of five of these cuvettes, with 0.9 ml of the control added to the other five cuvettes. Light output was again measured after 5 and 15 minute. A step-by-step protocol for the Microtox™ 90% Comparison Test is included as Appendix A (Azur Environmental, 1997).

Data analysis consists of comparing the light output from the bacteria in the control cuvettes with the light output from the sample cuvettes (Salazar, 1997). Using the control cuvettes to correct for the time dependent decrease in light emission by the bacteria, percent differences between the light outputs from control and sample replicates were quantified by averaging replicate values, and an overall toxicity value was determined. The definition of ‘toxicity’ used in this study is as defined by Azur Environmental (1997). A positive percent difference indicated that the sample was more toxic to the bacteria than the control. A negative percent difference indicated that the sample was less toxic to the bacteria than the control. Dutka and Kwan (1982) described negative differences as chemicals acting as growth stimulants and therefore enhancing light

production. Ranges of certainty (+/-), presented for each percent difference reported, were statistical values calculated to quantify the variance between replicate cuvettes of both controls and samples. Typically, an inhibitory response after 5 minutes indicates the presence of organics, while a response observed after 15 minutes can typically be attributed to the presence of metals (Microbics, 1992). Any test result for which wide ranges of certainty (high standard deviation) were obtained were re-tested to assure reproducibility of results. Quality control for the sensitivity of the bacteria used was routinely performed by measuring inhibition of a reference toxicant (10 mg/l phenol) (Salazar, 1997).

6.3.1.4 Nitrotox Testing Protocol

Nitrifier bioassay testing was conducted on WFS leachates, groundwater and lysimeter leachate samples, with three replicates for each sample. Nitrifying bacteria have been found to be especially sensitive to toxic compounds therefore making them ideal as sensitive test organisms (Salazar, 1997). An enriched culture of nitrifiers was grown in a 20-liter Plexiglas tank. To perform the bioassay, samples were pretreated with filtration and the pH adjusted to between 7.5 and 8.0. 500 ml of nitrifiers were withdrawn from the tank and washed twice with 80 mg/l NaNO_3 solution. The nitrifiers were then concentrated to 100 ml with a MLSS (Mixed Liquid Suspended Solids) concentration of around 10,000 mg/l. Aged tap water was used as the control. Twenty-five ml of each sample/control was placed in a 250 ml Erlenmeyer flask. Equal volumes of concentrated nitrifying bacteria suspension were added to each flask in 10 minute intervals. The MLSS concentration in the mixture was controlled at approximately 1500 mg/l. The flasks were then shaken at 200 rpm for one hour. Finally, 0.5 ml of ammonia substrate was added to the sample/control and the flask was shaken for two additional minutes. A Gilson Oxygraph 5/6 was used to measure the oxygen uptake rate (OUR). Inhibition percentages were calculated by comparing the OUR values of samples with the control sample.

6.3.1.5 Ion Chromatography Testing Protocol

Ion chromatography (IC) testing was performed on WFS leachates and groundwater and lysimeter leachate samples. WFS leachates were generated following the EP Water Test protocol. Ion chromatography testing was performed using a Dionex GP40 Ion Chromatograph. The test can be summarized as follows: An eluant, an aqueous carbonate-bicarbonate solution, carried the samples through a series of ion exchangers at a constant rate. Anions were separated according to their affinities for a low capacity, strong basic anion exchanger (columns). The separated anions were then subjected to a strongly acidic solution in the suppressor, which converted the anion to their highly conductive acid forms, and the eluant to a weakly conductive carbonic acid. Concentrations were measured by conductivity, and results were given in peaks of measured conductivities. The anion represented by each peak was identified based on retention time and the concentration quantified based on peak area or height (Salazar, 1997).

In order to identify and quantify the peaks obtained as results, a set of standards of known concentrations for the different anions suspected to be present in the samples were analyzed prior to testing, obtaining approximate retention times for each anion as well as peak-concentration ratios to generate calibration curves. As previously noted in section 6.3, similarities between easily quantifiable anion concentrations between WFS leachates and groundwater and lysimeter leachate samples could be used as indicators (tracers) of other leachable constituents such as organics and metals. A step-by-step protocol for IC testing is included as Appendix B.

6.3.1.6 Inductively Coupled Plasma Spectrophotometer Testing Protocol

Inductively coupled plasma (ICP) spectrophotometer testing was performed on WFS leachates, groundwater and lysimeter leachate samples using a Thermo Jarrell-Ash ICP. Leachates were generated following the appropriate EP Water Test or TCLP protocol. The emission source for the Thermo Jarrell-Ash ATOMSCAN 25 spectrometer is an

inductively coupled argon plasma spectrophotometer. A 2 kW crystal controlled radio frequency (RF) generator powers the source.

During plasma ignition, the gas stream is seeded with electrons from an external source. These electrons are accelerated in a torroidal path by the RF generator and collide with argon atoms to form more electrons and argon ions, which are in turn accelerated. This process continues until the gas becomes highly ionized (as a plasma), at which point the discharge is stable and self-sustaining.

Liquid samples are introduced into the plasma discharge as an aerosol suspended in argon gas. The sample aerosol stream passes through the center of the torroidal plasma discharge where it is desolvated, atomized, and the resultant elements are excited. Excited atoms that comprise the sample emit light at their characteristic wavelengths. This light is transmitted to the optical system where the light is dispersed by a diffraction grating and a narrow range of dispersed wavelengths fall onto a photomultiplier tube (PMT) detector. The detector converts light energy to electric current and the magnitude of the current is proportional to light intensity. The current is integrated over a predefined time period and this integrated change is measured by the detector circuit and passed on to the host computer which converts this value to concentration limits. A step-by-step protocol for ICP testing is included in Appendix C.

CHAPTER 7: RESULTS AND DISCUSSION

7.1 Geotechnical Results

For a detailed report on the geotechnical performance of the embankment materials used in this demonstration project see the companion geotechnical final report (Fox and Mast, 1998). From a geotechnical standpoint, the Auburn WFS performed well as a structural fill. The following information is provided as a brief summary.

Geotechnical laboratory tests conducted prior to embankment construction indicated the weathered Auburn WFS are best characterized as a silty sand, having good strength properties and a relatively low hydraulic conductivity. Samples of weathered WFS from the monofill had a fines content (i.e., passing #200 sieve) of 10 to 22%. These values fall within the range of 6% to 40% fines for WFS samples taken from several Indiana foundries. Laboratory Proctor compaction tests indicated that, similar to natural soils, the maximum dry unit weight and optimum moisture content (OMC) for the WFS increases and decreases, respectively, with increasing compaction energy (Figure 8). A vibratory compaction test indicated that vibration was not effective in compaction WFS. Values of California Bearing Ratio (CBR) were 17 and 18 for soaked and unsoaked specimens, respectively, indicating that the WFS is a “fair” material for use as a base coarse or subgrade material. The WFS specimen experience no volumetric swelling during the CBR soaking test. Flexible-wall hydraulic conductivity tests showed that depending on compaction moisture content and compaction energy, the hydraulic conductivity of the WFS ranged from 1×10^{-8} to 7×10^{-7} m/s. Direct shear tests on dry WFS specimens gave an angle of internal friction of 35° to 38° and a small cohesion intercept.

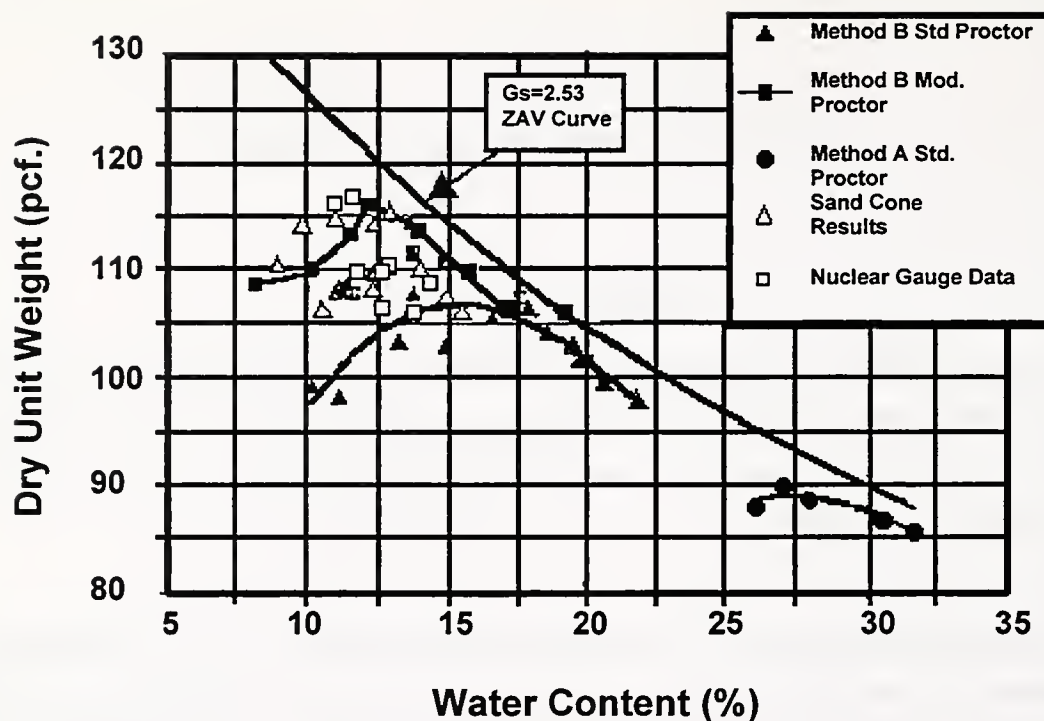


Figure 8 – Field and Laboratory Compaction Testing of Auburn WFS

The pre-construction test pads showed that a heavy rubber-tire roller (36,000 kg) was more effective than a smooth-drum roller in compacting WFS and that significant watering was not needed to reach OMC. As a result of these field tests, INDOT required that the WFS be laid down in 200 mm lifts with a moisture content between 12% and 15%.

Each lift was then to receive a minimum of 6 passes of the heavy rubber-tire roller. Using this method specification, field compaction proceeded with few problems. The WFS compacted tightly, with no observed pumping or rutting under the wheels of heavy construction equipment. The only problems encountered during construction were foreign objects in the WFS (e.g., slag, cores, discarded metal castings, and welding rods) and airborne dust. These problems were solved by pushing foreign objects into the fill

with a blade and frequent watering of the working surface. In general, foundry operators who wish to reuse their WFS for construction should not incorporate foreign objects or bag house dust into their WFS stream.

Figure 8 presents field compaction data taken during embankment construction along with standard and modified Proctor compaction curves obtained in the laboratory. The field data generally lie between the two laboratory curves, indicating that the ASTM Method B Proctor tests provided good indications of the WFS unit weight when the WFS were compacted according to the INDOT method specification. A comparison of measurements obtained using the sand cone, nuclear density gage (NDG), and Speedy Moisture Meter (SMM) indicated that the SMM was generally more reliable than the NDG for the measurement of moisture content in the field. However, dry unit weights measured using the NDG were in good agreement with those obtained using the sand cone method. Thus, the NDG may be useful for quality control in the field provided a suitable correction factor is developed for moisture content.

Horizontal inclinometer and settlement plate measurements indicated a maximum foundation settlement of 45 mm. Net compression of the embankment material was much less. Subtracting the settlements of the upper and lower plates for each settlement plate nest the following values for net embankment compression are obtained: clay borrow, 7 mm; natural sand, 7 mm; and WFS, 3 mm. Figure 9 shows the change in lateral displacement since 8 August 1996 as a function of depth for the east vertical inclinometer. The maximum lateral movement within the WFS was 11 mm. The west vertical inclinometer generally showed smaller lateral displacements, except for the final reading on 6 June 1998 which showed that a movement of about 10 mm occurred during spring 1998. In general, the WFS performed as well or better than the natural sand and clay fill materials used for this project, although deformations have been small for all materials.

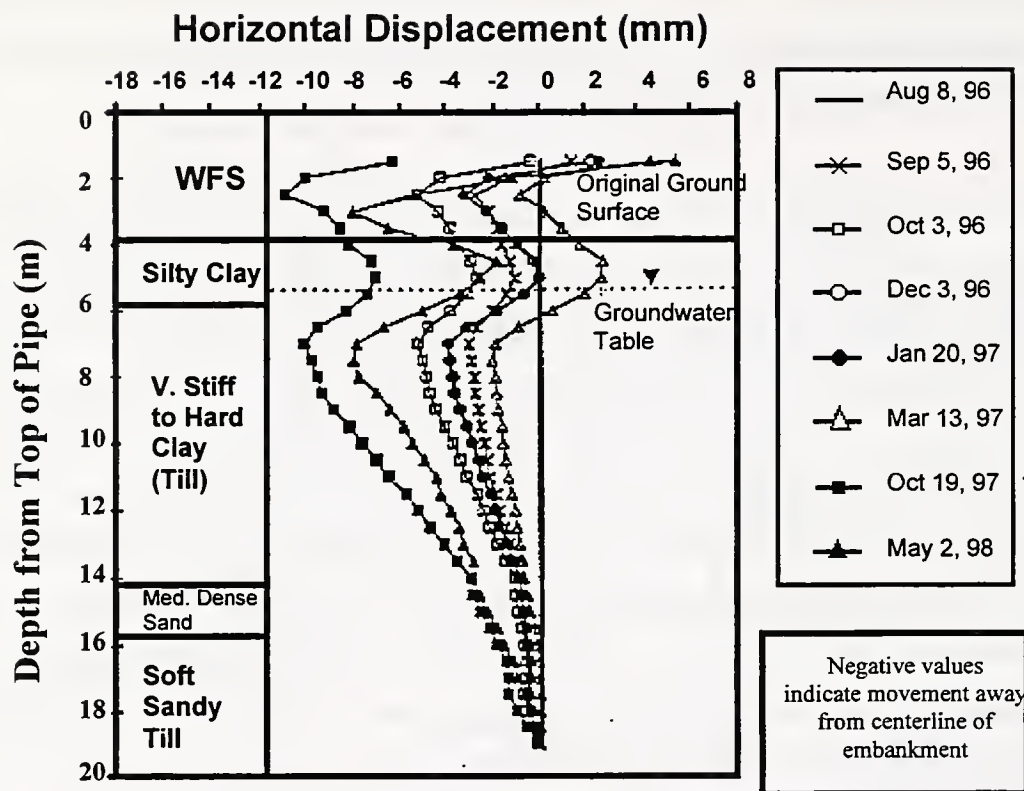


Figure 9 – Horizontal Displacement of East Vertical Inclinometer

The results of the sealed double-ring infiltrometer (SDRI) test showed that a steady state inflow rate of 0.0078 m/s was reached within a 5-week period. The corresponding hydraulic conductivity is 1×10^{-7} ml/s. This value is consistent with field observations of low hydraulic conductivity made in a similar WFS embankment project in Wisconsin (Lovejoy, 1996). In addition, the estimated hydraulic conductivity value falls within the range of values obtained from the laboratory tests. These results indicate that compacted Auburn WFS are not a freely draining material. This may have important implications for the design of drainage systems and the stability of embankments using WFS by-products.

Soil borings in the completed embankments indicated the following range of blow counts for the different materials: WFS, 25 to 54; natural sand, 18 to 50; clay, 16 to 37. These

values suggest the compacted WFS had relative densities which were higher than the natural sand. In addition, the WFS had substantially higher blow counts than the clay borrow.

7.2 Environmental Results

7.2.1 Introduction

Statistical analysis of environmental data was performed using the SAS statistical packages installed at the Purdue University Computing Center and the STATISTIX for Windows Analytical Software. Analysis of Variances (ANOVA) full and partial factorial analysis and various comparison methods were used to analyze the data.

7.2.2 Microtox™ Testing

ANOVA of Microtox™ T5 (5 minute) and T15 (15 minute) inhibitions were statistically equal for both the WFS stockpile and job site leachate samples, but were not equal to each other. Job site samples are believed to be more representative of the WFS embankment as the samples were obtained over the entire depth of the monofill. INDOT has subsequently developed a stratified sampling protocol (Appendix D) to ensure representative sampling of a WFS stockpile. In short, the user must ensure stockpile sampling and testing is representative of the WFS for the intended reuse application.

Groundwater wells and lysimeters leachates were determined to be statistically equal by location and replicate, but were statistically different over time. Microtox™ inhibition (toxicity) versus sampling date for T5 and T15 tests are shown in Figures 10-13 in combined and separated (i.e., wells and lysimeters separated) formats. Plots in the combined format are based on 'best fit' data while plots in the separated format are spline data points. INDOT's *Acceptance Criteria for Waste Foundry Sand Reuse Based on the*

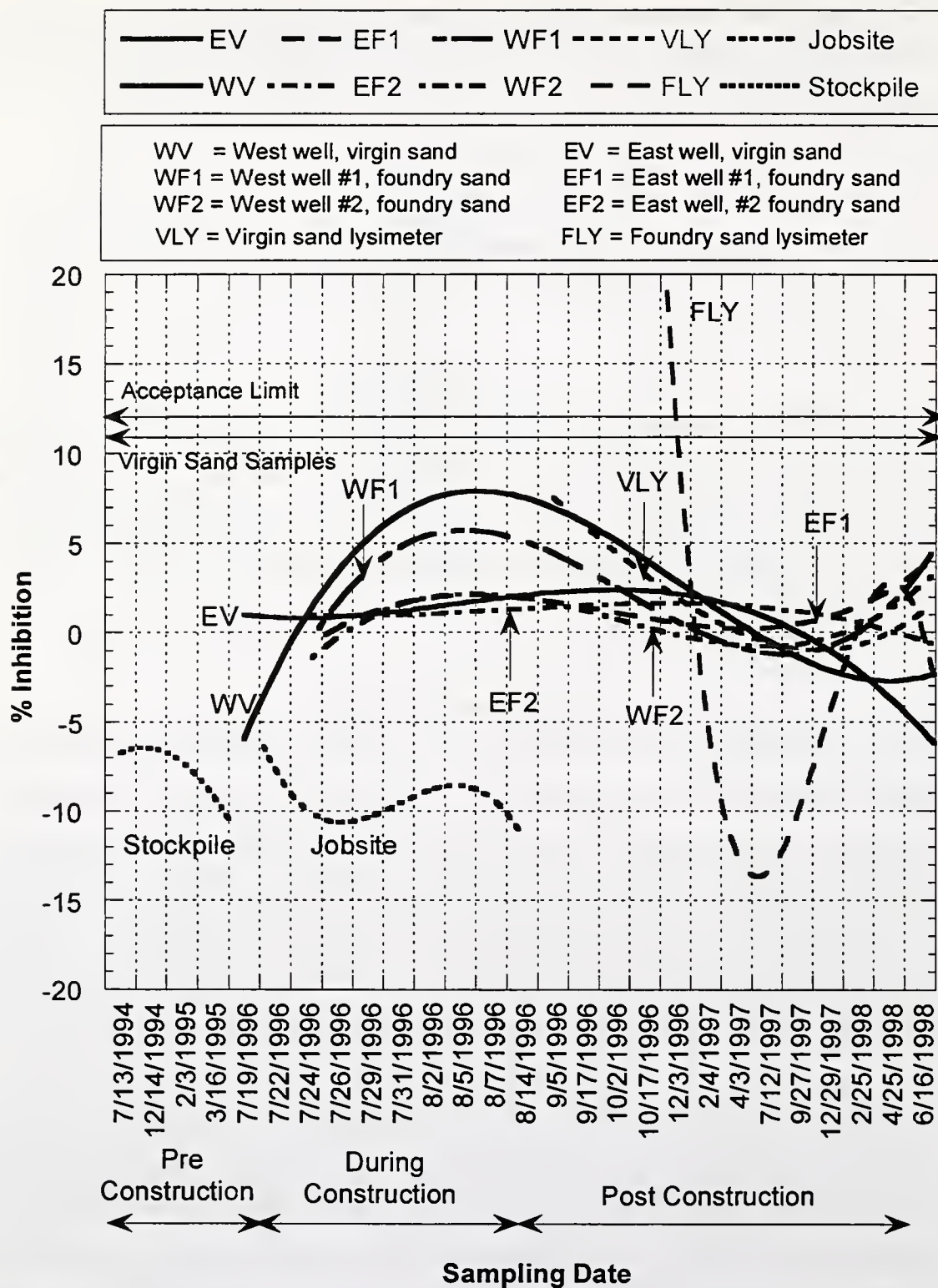


Figure 10 – T5 Microtox™ Inhibition (Toxicity)

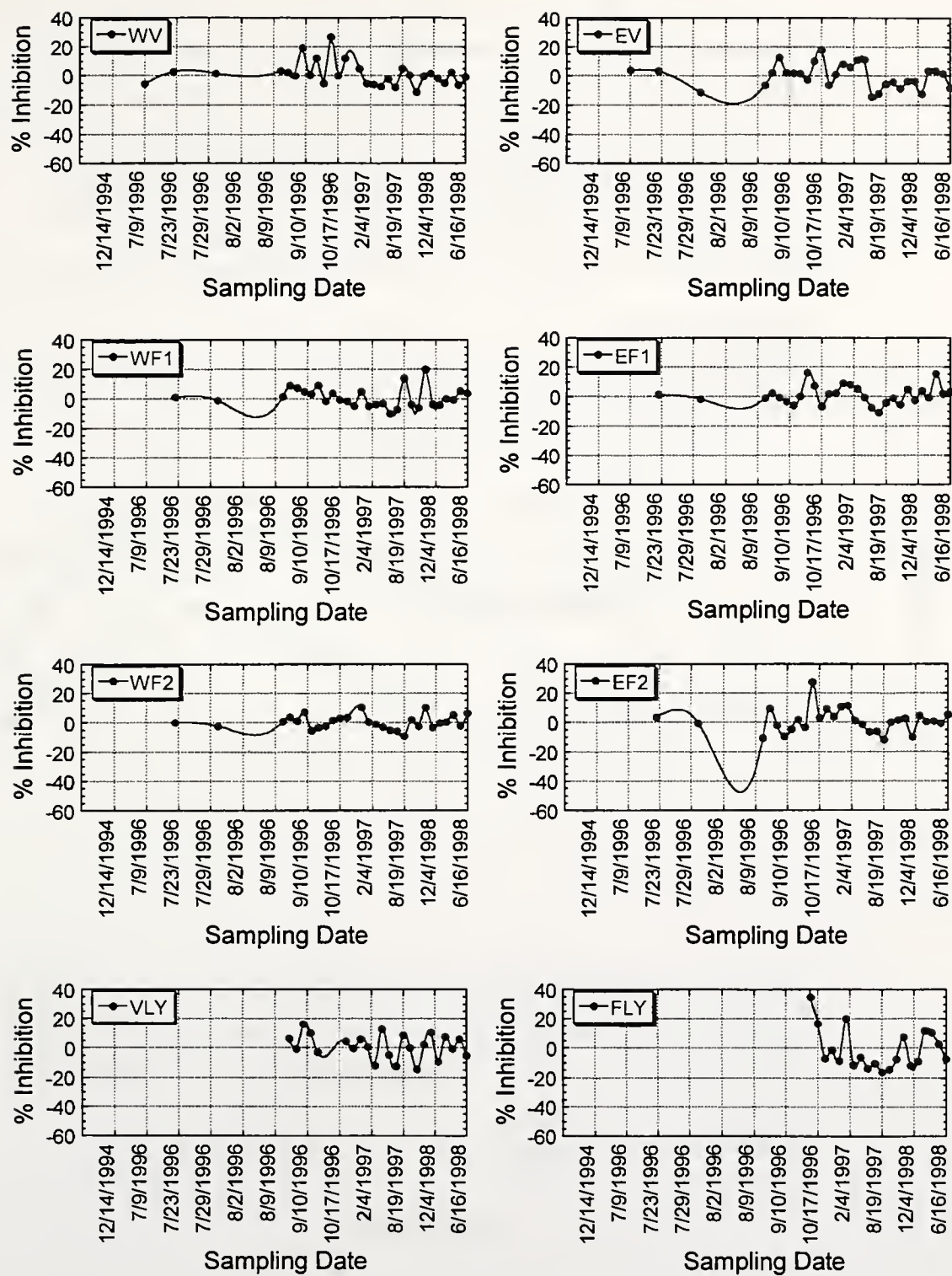


Figure 11 – T5 Microtox™ Inhibition (Toxicity)

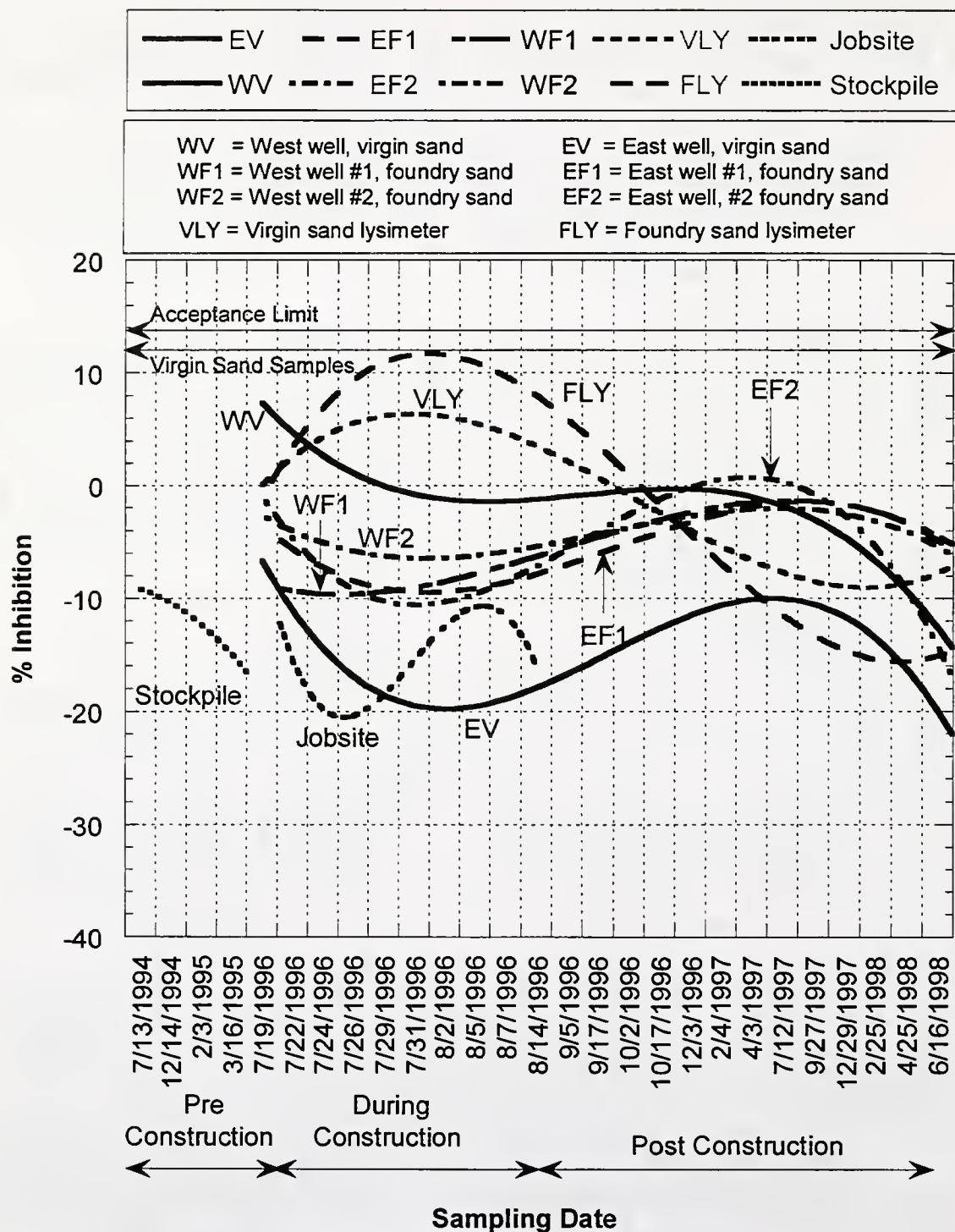


Figure 12 – T15 Microtox™ Inhibition (Toxicity)

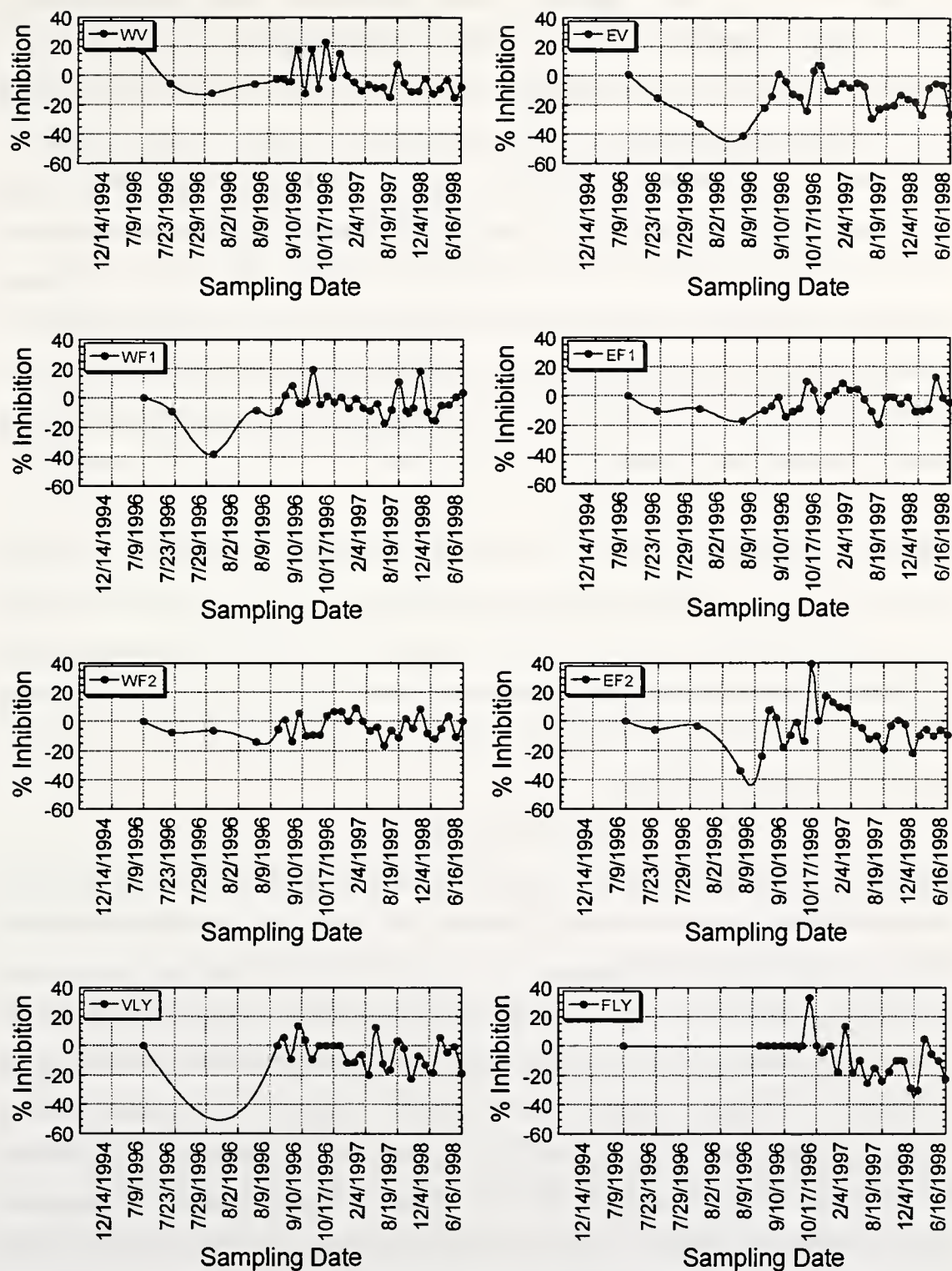


Figure 13 – T15 Microtox™ Inhibition (Toxicity)

*Microtox*TM *Bioassay Test* (Appendix E), based on previous testing of unused foundry sands and typical highway construction sands, has established an acceptance limit of 12% inhibition for both the T5 and T15 tests. Consequently, *Microtox*TM test results of the groundwater and natural sand lysimeter leachate samples have consistently exhibited inhibitions below the acceptance limit, as well as below inhibitions typical of natural virgin sands. When including WFS job site leachate data in the analysis, *Microtox*TM T15 results indicate the WFS did have an impact on the EV well and FLY, although inhibitions were typically negative indicating no toxicity. Negative inhibitions indicate that samples are non-toxic and may enhance bacterial activity. This is a strong indication that potential contaminants in the WFS would not result in inhibition levels higher than those for common natural sands.

Salazar (1997) proposed *Microtox*TM inhibition limits of less than 10% as nontoxic and between 10%-25% as slightly toxic, based on review of the literature and historical EC_{50} values. As previously noted, an EC_{50} value is the concentration of a compound causing a 50% reduction of the light omitted by the bacteria over a given exposure time. Using this convention, the WFS are considered nontoxic and would not result in a negative environmental impact to the site.

It should be noted, an increase in *Microtox*TM T5 inhibitions for the FLY samples were observed for samples tested between 2 October 1996 to 17 October 1996. *Microtox*TM inhibition values for the FLY peaked on 2 October 1996 with an inhibition value of 75.6% and decreasing thereafter. Using Salazar's convention, these samples would be considered moderately toxic to very toxic. East down-gradient wells, while following the same trend as the FLY samples remained in the 'non-toxic' category probably due to dilution with the groundwater. The VLY samples also displayed a similar trend of increasing inhibition, but approximately two weeks earlier (peaking on 10 September 1996). Inhibitions remained in the 'non-toxic' category, probably due to the freely draining characteristics of the natural sand.

The increase in FLY inhibitory values could be attributed to one of two events: first, the possibility of a 'first flush' effect when the highest concentration of contaminants would be expected. A first flush refers to the first quantity of water to pass through the embankment, which according to laboratory observations would contain the highest concentrations of soluble pollutants. Secondly, an inhibitory contaminant may have been leached from the HDPE geomembrane or the PVC slotted pipe used to construct the lysimeters. While both lysimeters were constructed using the same materials, the inhibitory effect may only have been detected in the FLY due to the limited amount of water available to remove soluble contaminants from these plastics. The VLY, a more freely draining material, may have had enough water passing through it such that soluble contaminants were diluted, or passed before the first sample was collected (Salazar, 1997). This may be reflected in the earlier, less pronounced, increase in VLY leachate inhibitions. This second hypotheses is likely to be the most probable as subsequent laboratory testing of leachate from the geomembrane exhibited a similar toxic response to the bacteria. Furthermore, the WFS in the monofill was weathered for several years, and thus the first flush likely would have occurred before the sand was transported to the site.

7.2.3 Nitrotox Testing

ANOVA of Nitrotox inhibition test results indicate no significant difference between up- and down-gradient wells and lysimeters although there was a statistical difference with time. Figures 14 and 15 display Nitrotox measured inhibition versus sampling date. Test results did not indicate a similar response, as with the Microtox™ test, to leaching of a contaminant from the geomembrane. This may be a result of the nitrifying bacteria not being sensitive to the particular contaminant(s). When including the WFS job site leachate data in the analysis, the Nitrotox test results indicate the WFS did have an impact, primarily on the EV well and FLY. WFS job site leachate inhibition, however, were typically negative over time indicating no toxicity. Well and lysimeter Nitrotox inhibitions were typically less than 40%; generally Nitrotox inhibitions less than 50% are considered non-toxic (Salazar, 1997). As with the Microtox™ bioassay test results,

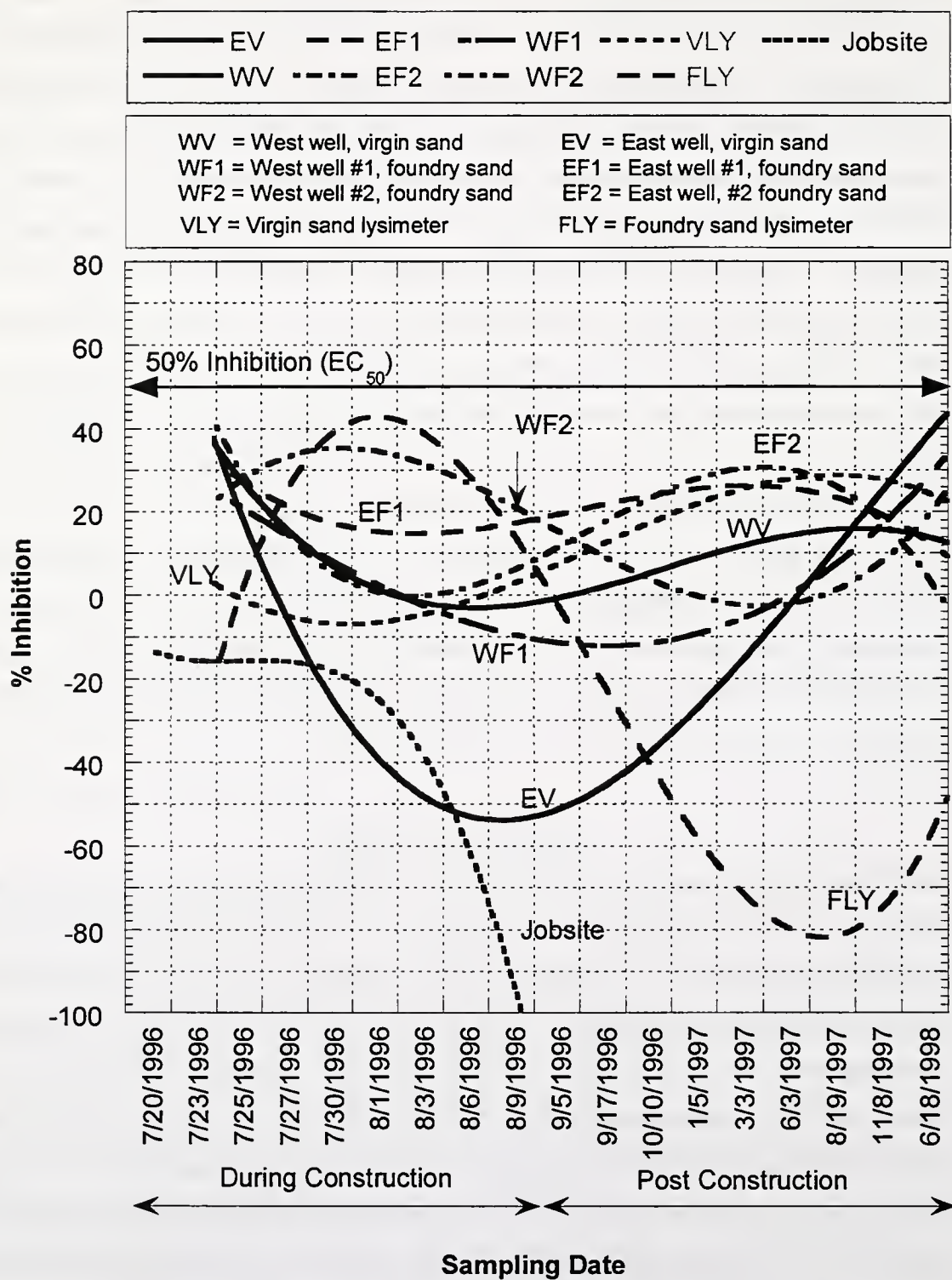


Figure 14 – Nitrotox Inhibition

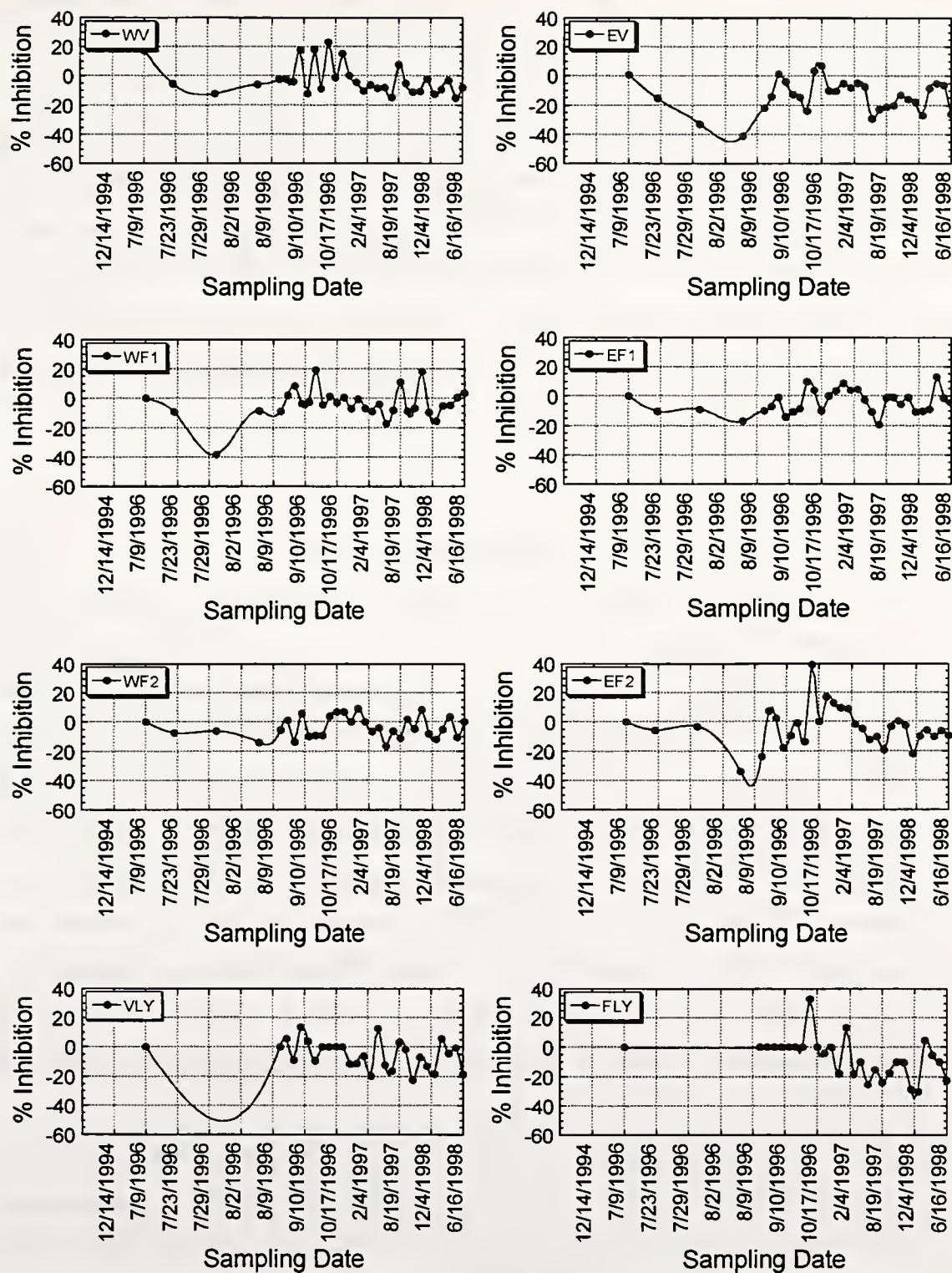


Figure 15 – Nitrotox Inhibition

Nitrotox results indicate these WFS will not likely result in a negative environmental impact on the site.

Attempts to correlate Microtox™ inhibition with Nitrotox inhibition were unsuccessful. The maximum coefficient of determination ($R^2=0.11$) was obtained between the Microtox™ T15 and Nitrotox data for the WFS job site leachate data. Previous research (Blum and Speece, 1991) found the Microtox™ test was as sensitive as the nitrifying bacteria *Nitrosomonas* and fat head minnows. They found a good correlation ($R^2=0.70$ to 0.82) between Microtox™; three bacterial groups, aerobic heterotrophs, *Nitrosomonas*, and methanogens; and the fat head minnow.

7.2.4 Ion Chromatography Testing

ANOVA of ion concentrations (F^- , Cl^- , SO_4^{2-} , and NO_3^- -N) indicated the following:

- F^- concentrations were statistically different by location and time. WFS job site leachate concentrations and EV well and FLY concentrations were highest with the highest concentration (9mg/l) measured in the FLY (3 June 1997). West up-gradient wells exhibited the lowest F^- concentrations. Figures 16 and 17 display F^- concentration by sampling date. While fluoride transport from the WFS into the EV well and FLY is indicated, no F^- concentration exceeded the IDEM beneficial reuse criteria of 14.0 mg/l (for a Type III classified waste) with most values below the more conservative Type IV criteria (1.4 mg/l). Waste parameter concentrations below the Type III criteria are acceptable for beneficial reuse in Indiana. Similarly, F^- concentrations were consistently below 1996 drinking water standards (DWS) in both the up- and down-gradient wells.
- Cl^- and NO_3^- -N concentrations were not significantly different by location but were statistically different over time. WFS job site leachate Cl^- and NO_3^- -N concentrations were lower than respective concentrations found in the up- and down-gradient wells. Cl^- and NO_3^- -N concentrations, by sampling date, are displayed in Figures 18 and 19 and Figures 20 and 21, respectively. Cl^- concentrations were significantly below the

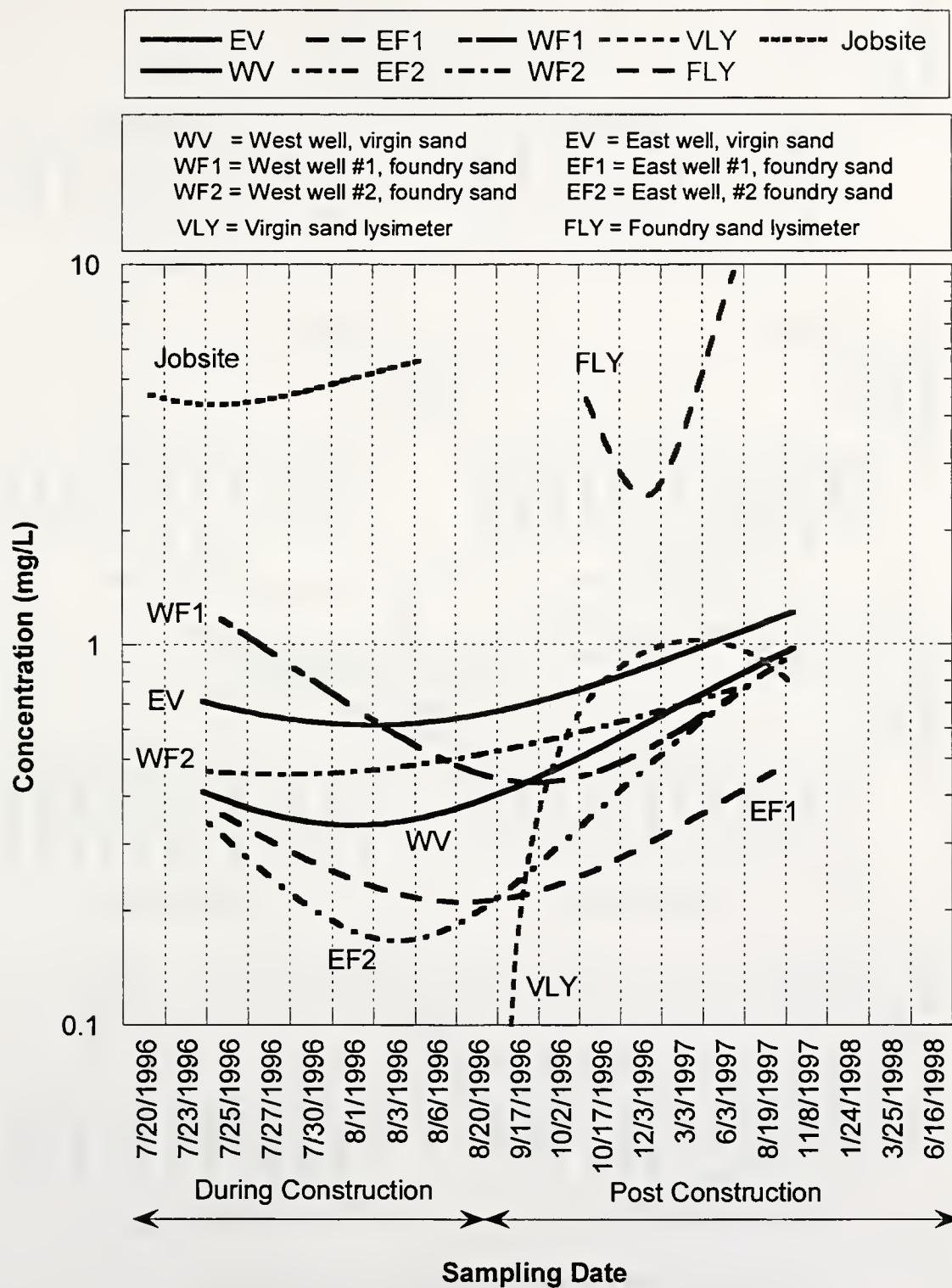


Figure 16 – Ion Chromatography (IC) for Fluoride (F^-)

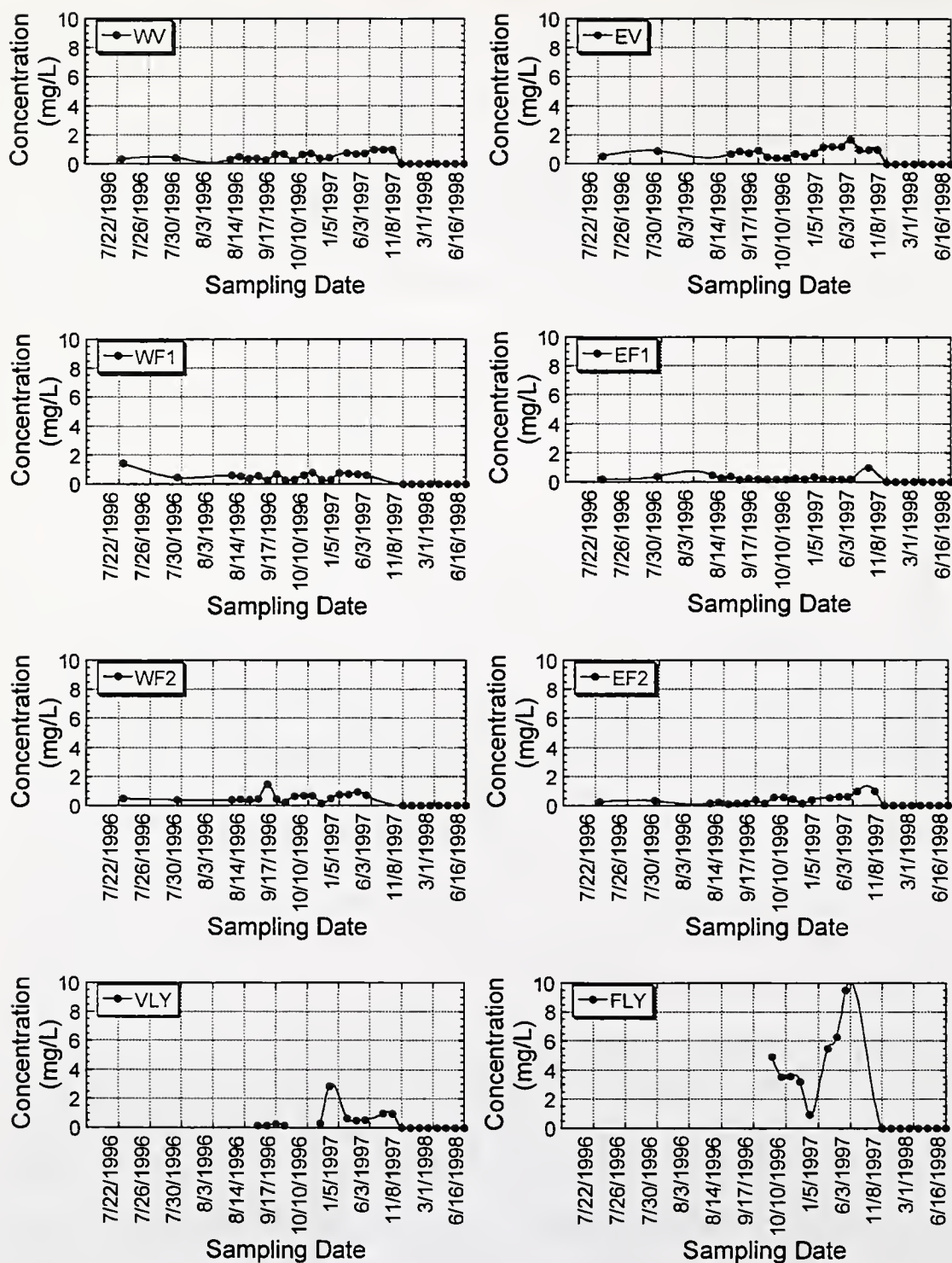


Figure 17 – Ion Chromatography (IC) for Fluoride (F)

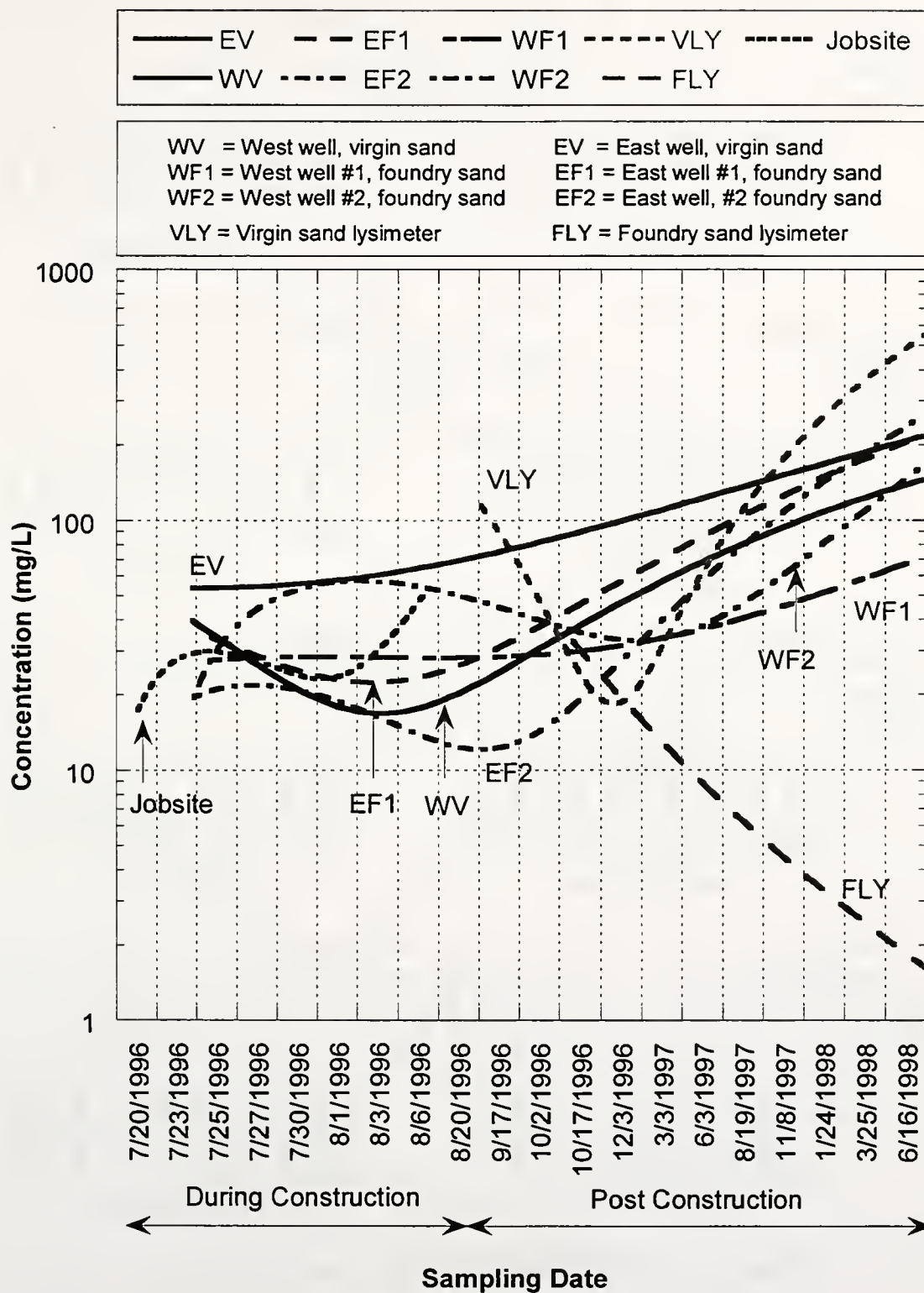


Figure 18 – Ion Chromatography (IC) for Chloride (Cl⁻)

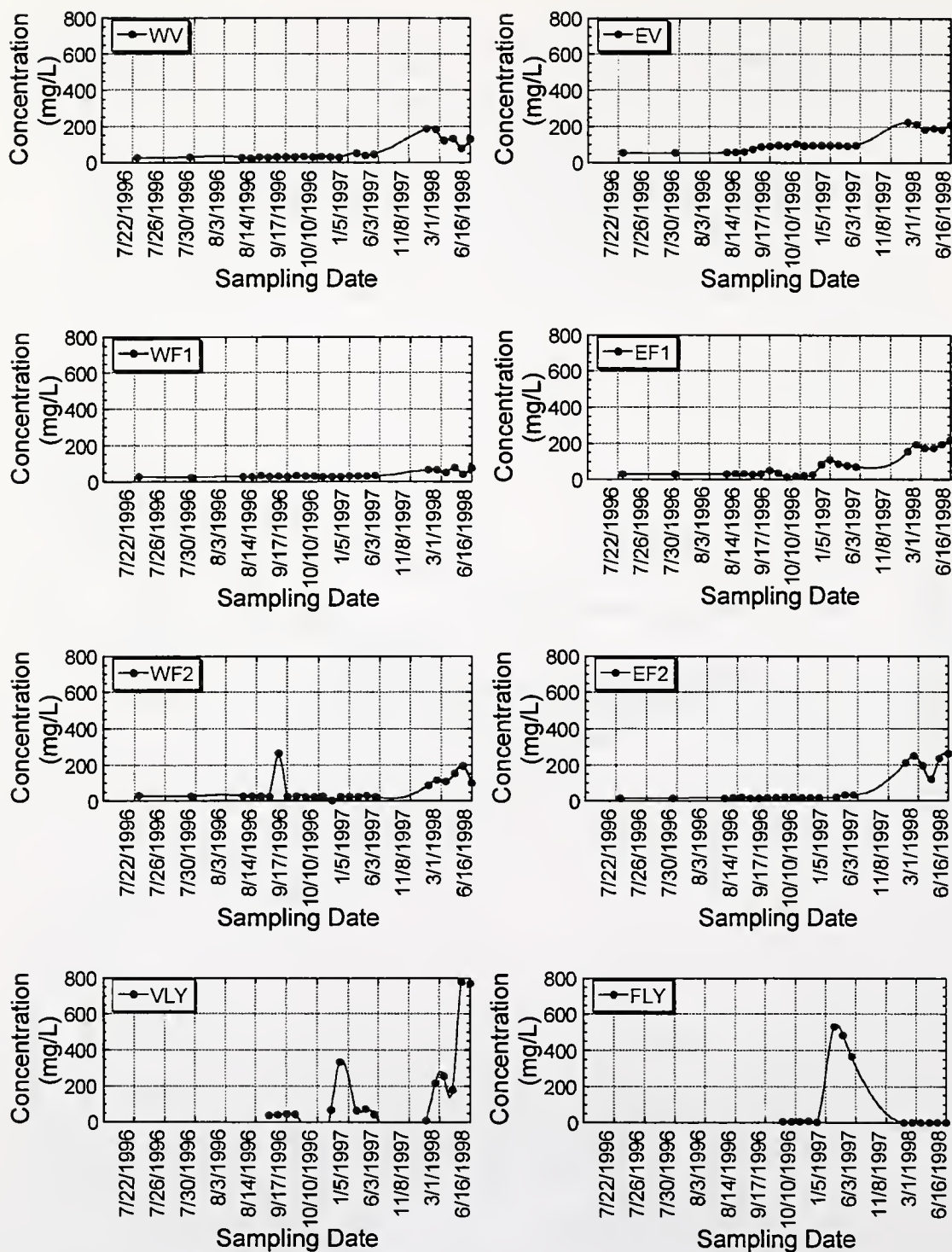
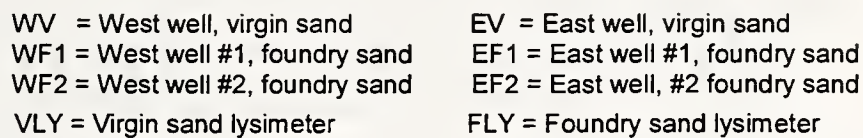


Figure 19 – Ion Chromatography (IC) for Chloride (Cl⁻)



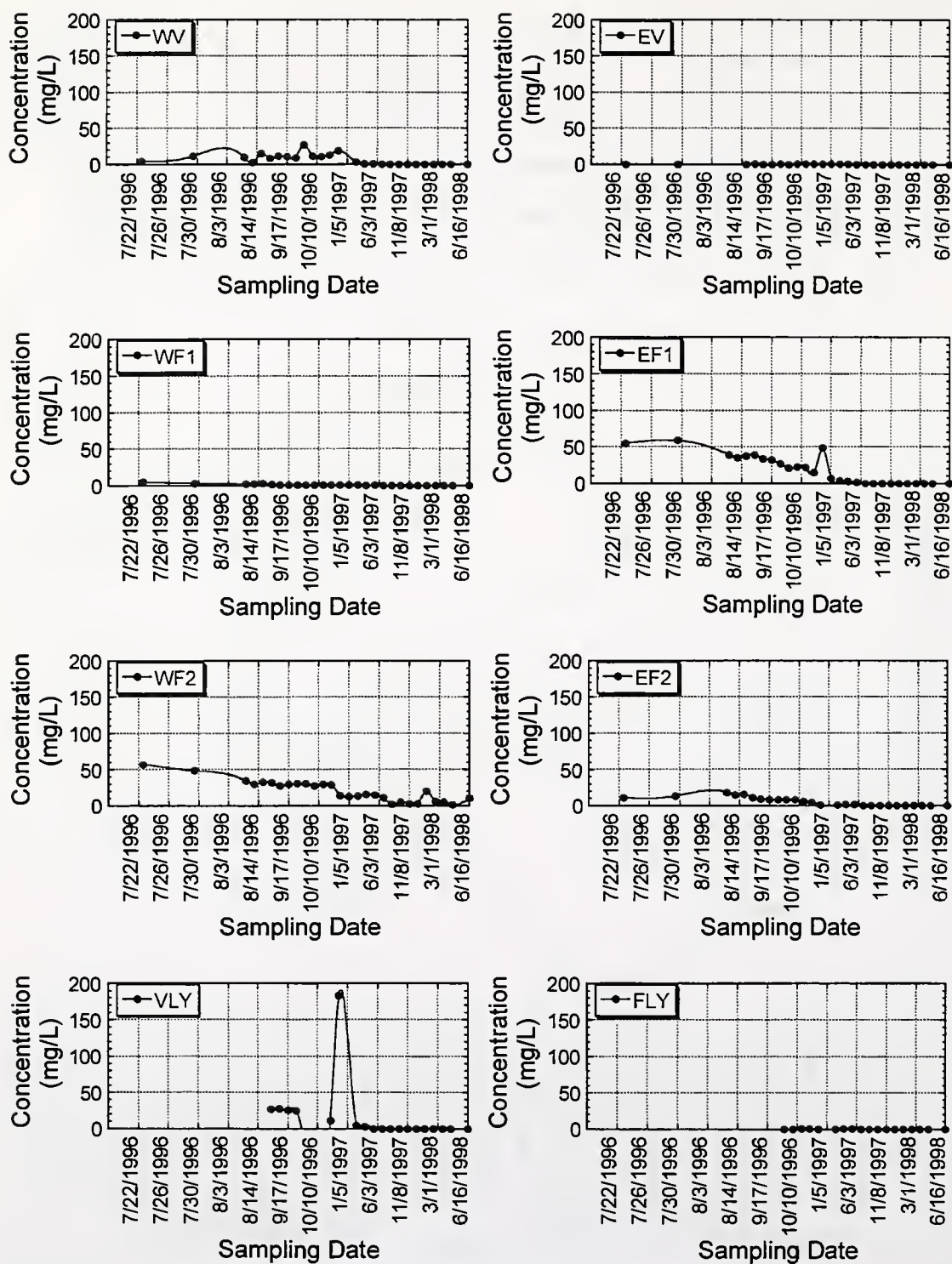


Figure 21 – Ion Chromatography (IC) for Nitrate (NO_3^- -N)

IDEM Type III criteria of 2,500 mg/l and generally below the Type IV criteria (25 mg/l). Cl^- well concentrations were below DWS except for two samples, one each in the up-gradient WF2 well and the down-gradient EF2 well, although concentrations barely exceeded the DWS of 250 mg/l (MCL). NO_3^- -N concentrations appear inherent in the groundwater, probably from farmland runoff, and not a result of the WFS. Nitrate (as N) concentrations exceeded DWS.

- $\text{SO}_4^{=}$ concentrations were highest in the WFS job site leachates and east down-gradient wells and FLY. Highest concentrations were found in the FLY. West up-gradient wells exhibited the lowest sulfate concentrations. $\text{SO}_4^{=}$ concentrations versus sampling date are shown in Figures 22 and 23. Subsequent to March 1997, $\text{SO}_4^{=}$ concentrations have varied significantly by location and were statistically different over time throughout the post construction period (15 August 1996 – 16 June 1998). While sulfate transport from the WFS into the east wells and FLY is indicated, no $\text{SO}_4^{=}$ concentration exceeded the IDEM beneficial reuse criteria of 2500 mg/L (for a Type III classified waste). Well $\text{SO}_4^{=}$ concentrations did exceed DWS in some instances, primarily in the down-gradient wells.

As previously noted, ions were intended to act as ‘tracers’ to substantiate the migration of potential contaminants (e.g., soluble organics) from the WFS embankment into the lysimeters and groundwater wells, thereby further substantiating bioassay inhibition results. IC results indicate F^- and $\text{SO}_4^{=}$ are indeed migrating from the WFS into the FLY and subsequently into down-gradient well(s), although at more dilute concentrations. IC results indicate no negative environmental impact to the site from use of the WFS as embankment material.

7.2.5 Inductively Coupled Plasma Spectrophotometer Testing

Metal concentrations in wells and lysimeters, except for Cd and Mn, were below IDEM Type IV and Type III beneficial reuse criteria. Ag (≤ 0.05 mg/L), Ba (≤ 1.0 mg/L), and Ni (≤ 0.2 mg/L) concentrations were below IDEM Type IV criteria (shown in

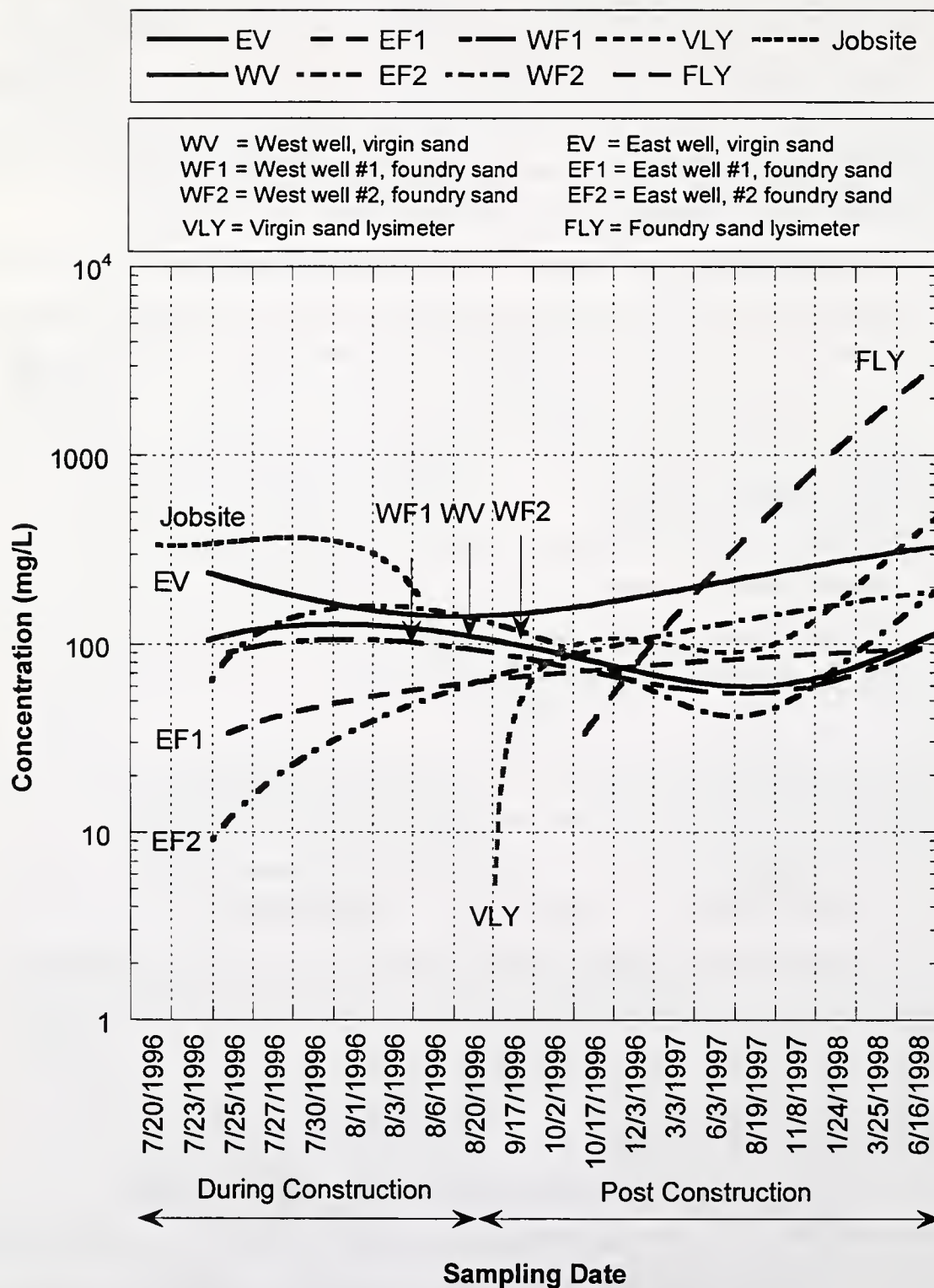


Figure 22 – Ion Chromatography (IC) for Sulfate (SO_4^{2-})

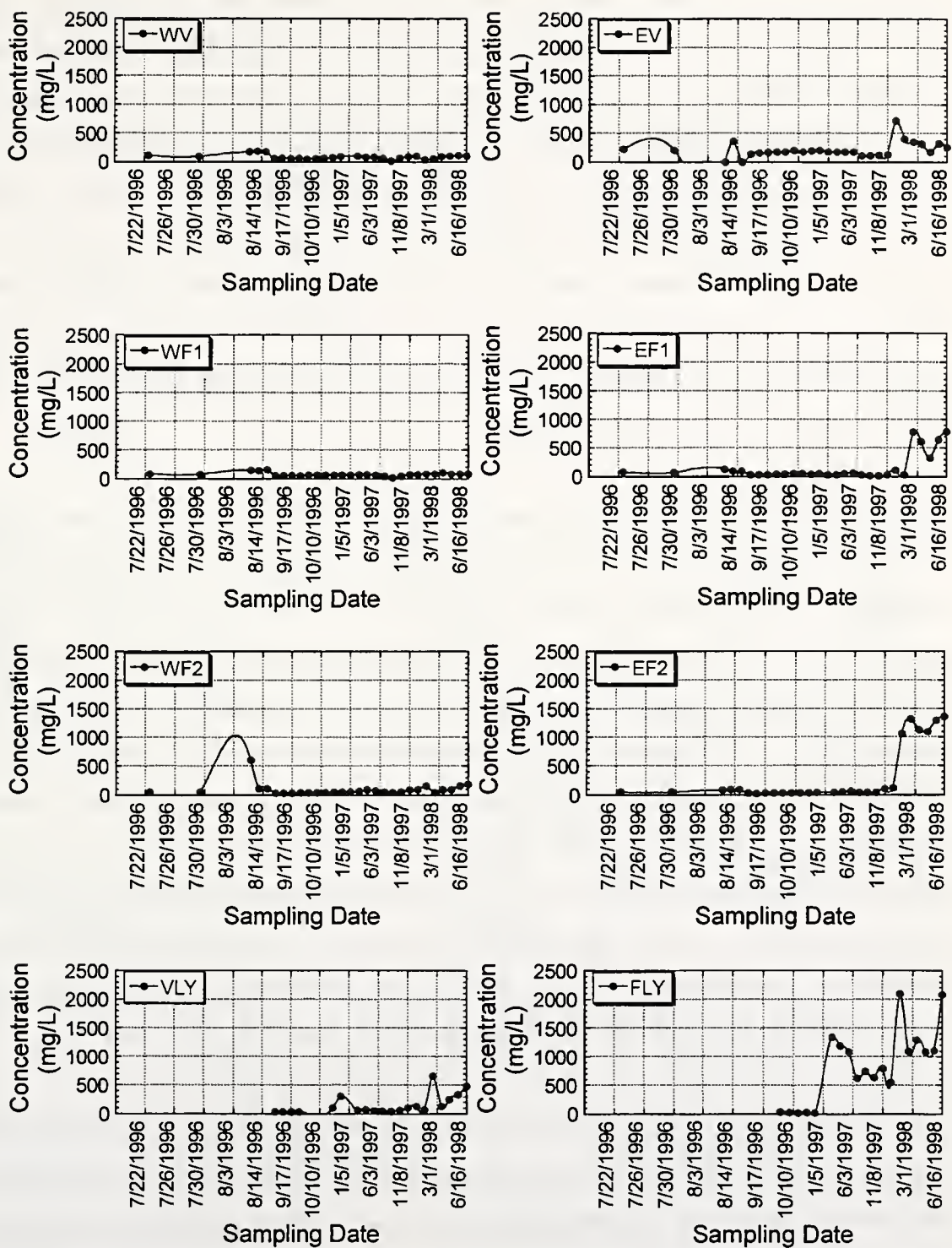


Figure 23 – Ion Chromatography (IC) for Sulfate (SO_4^{2-})

parenthesis). As (≤ 0.5 mg/L), Cr (≤ 0.5 mg/L), Cu (≤ 2.5 mg/L), Pb (≤ 0.5 mg/L), Se (≤ 0.1 mg/L), and Zn (≤ 25.0 mg/L) were below IDEM Type III criteria. Metal well and lysimeter concentrations typically were not statistically different by location suggesting no site impact from the WFS. Metal concentrations versus sampling date for the metals Ag, Ba, Ni, As, Cr, Cu, Pb, Se, and Zn are shown in Figures 24-41, respectively. The same data for each metal is shown in both combined and separated (i.e., wells and lysimeters separated) formats. Plots in the combined format are based on 'best fit' data while plots in the separated format are spline data points. WFS job site leachate metal concentrations were consistently below IDEM Type III criteria and except for Mn, Pb, and Se were below IDEM Type IV criteria.

Cd concentrations (17% of samples tested) exceeded IDEM Type III beneficial reuse criteria (≤ 0.1 mg/l) in both up- and down-gradient wells. Cd concentrations were statistically equal between up- and down-gradient wells. Cd concentrations however, were negligible in lysimeter samples and WFS job site leachate samples. Cd appears inherent to the background water quality of the site and not a result of a negative impact from the WFS. Cd concentrations versus sampling date are shown in Figures 42 and 43. Similarly, Mn well concentrations exceeded IDEM Type III beneficial reuse criteria (≤ 0.5 mg/L) in both up- and down-gradient wells and lysimeters (36% of the samples tested), but were statistically equal by location, suggesting Mn was present in the existing groundwater and not a result of Mn leaching from the WFS. Furthermore, WFS job site leachate Mn concentrations were consistently below IDEM Type III beneficial reuse criteria. Mn concentrations versus sampling date are shown in Figures 44 and 45.

It is interesting to note the similarity (impact) of the WFS on the FLY and EV well from various test results (e.g. Microtox™ and Nitrotox inhibitions, IC fluoride concentrations). When the project site was designed, the B-borrow (natural sand) embankment was placed adjacent (north) of the WFS embankment to act as a control section, believing the natural sand would more closely simulate the WFS performance. In reality, the hydraulic

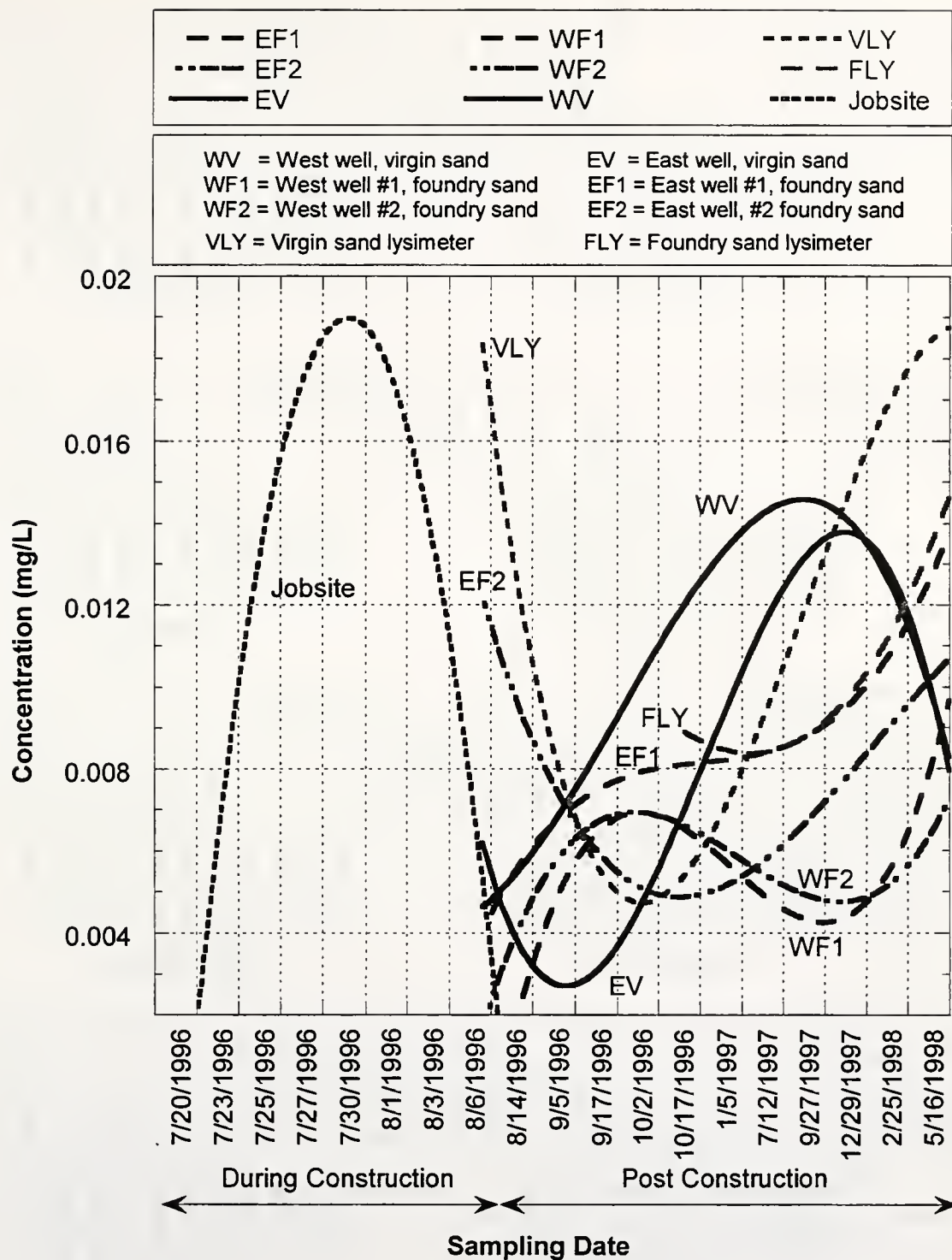


Figure 24 – Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Ag

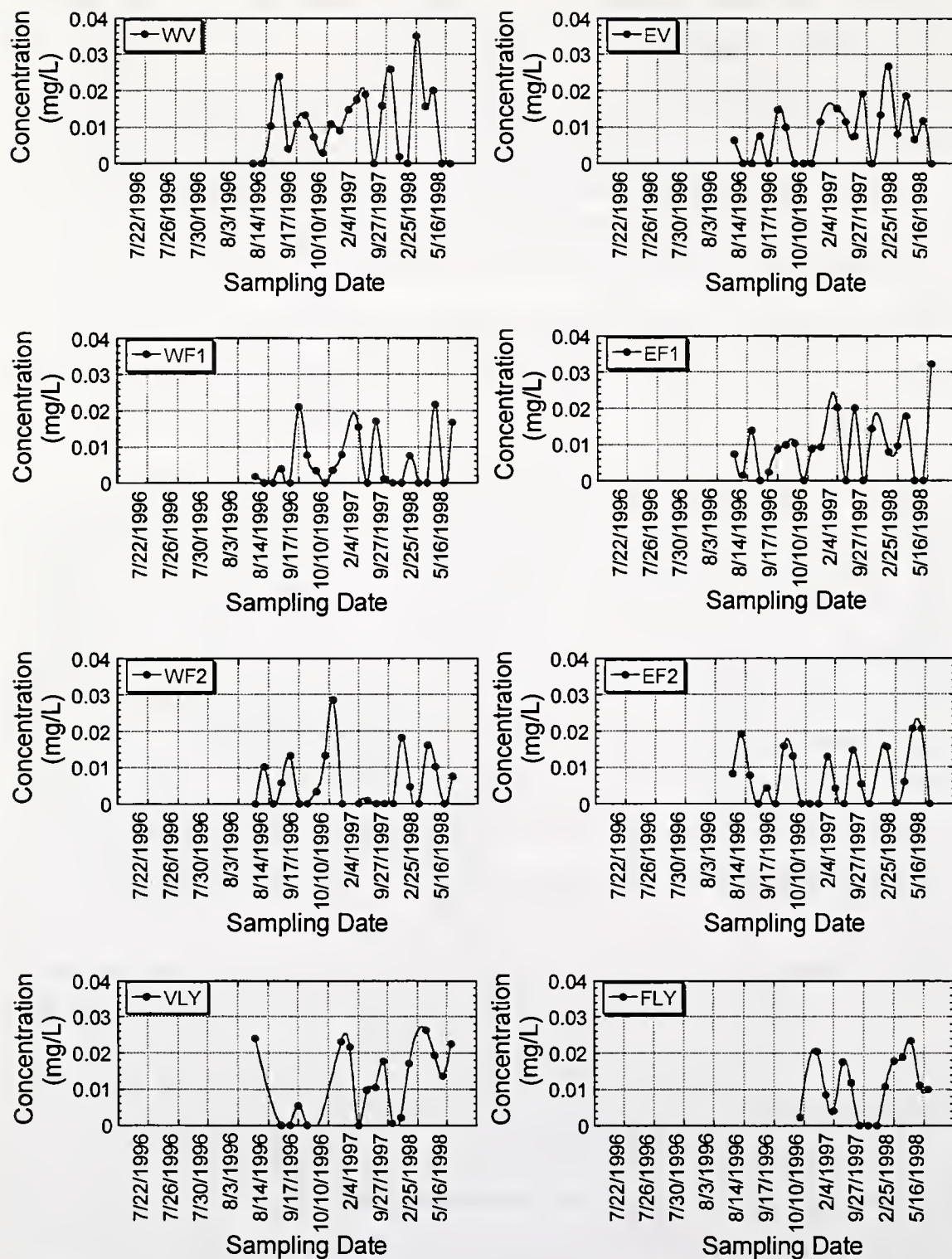


**Figure 25 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Ag**

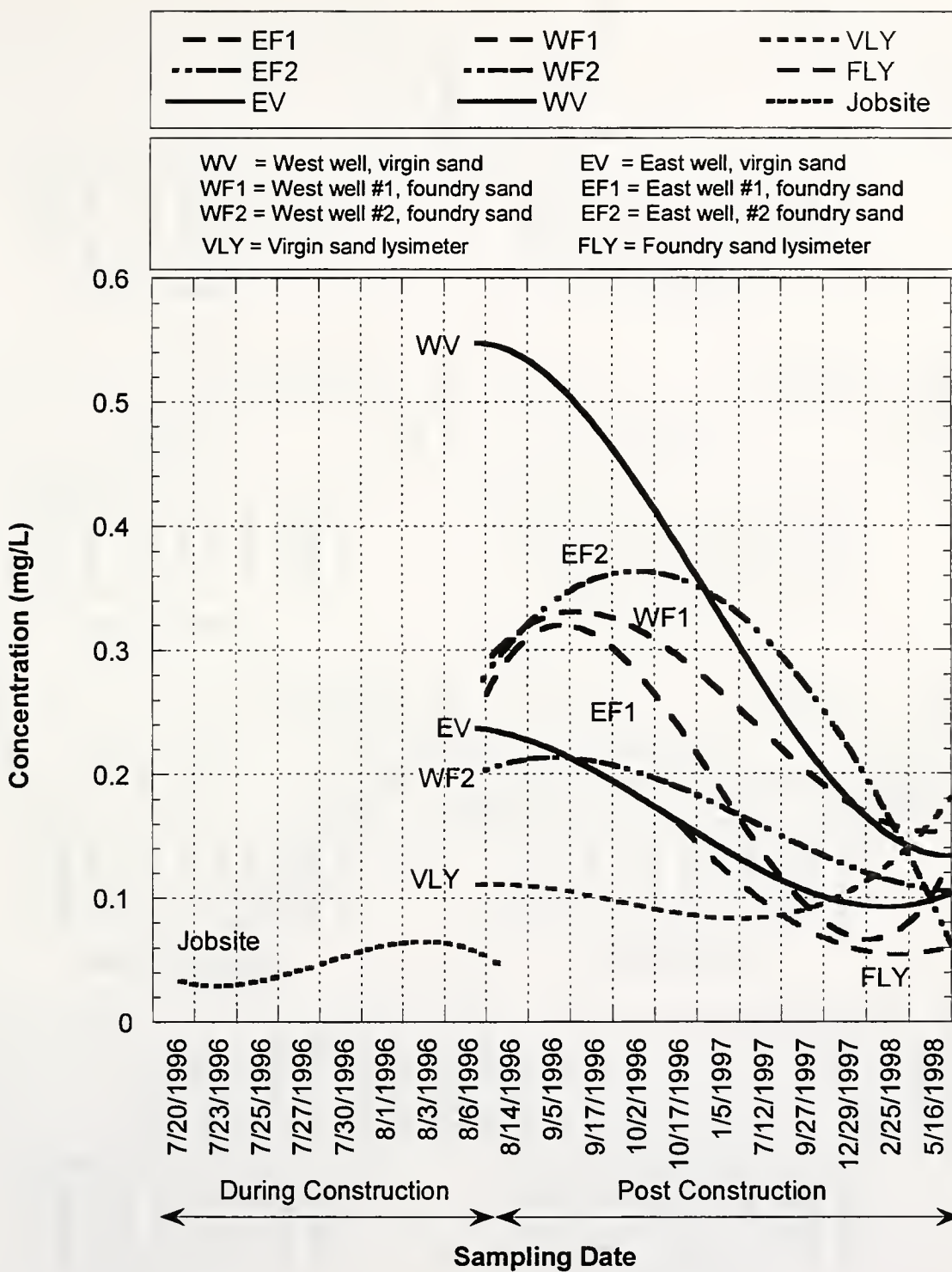


**Figure 26 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Ba**

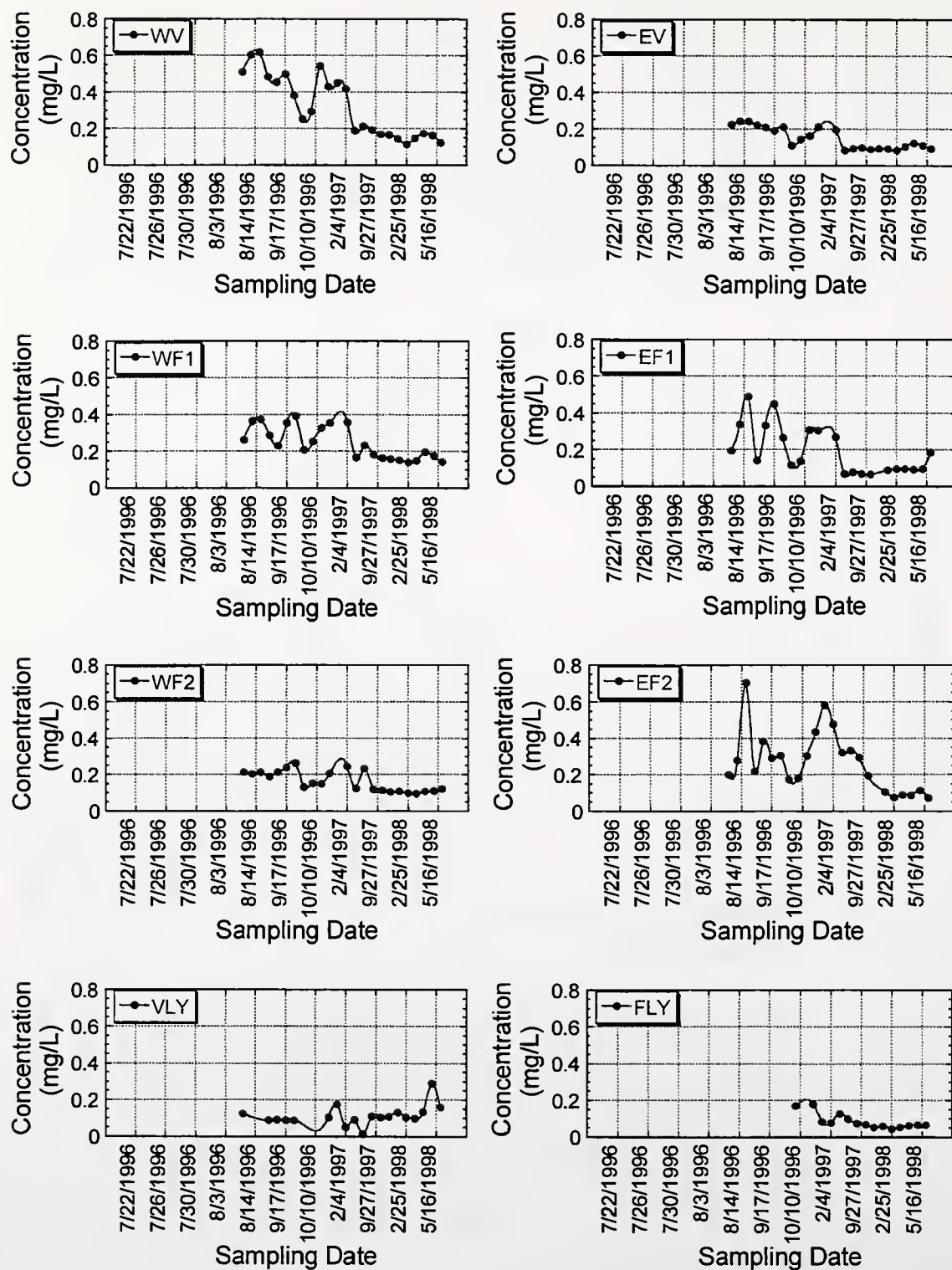


Figure 27 – Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Ba

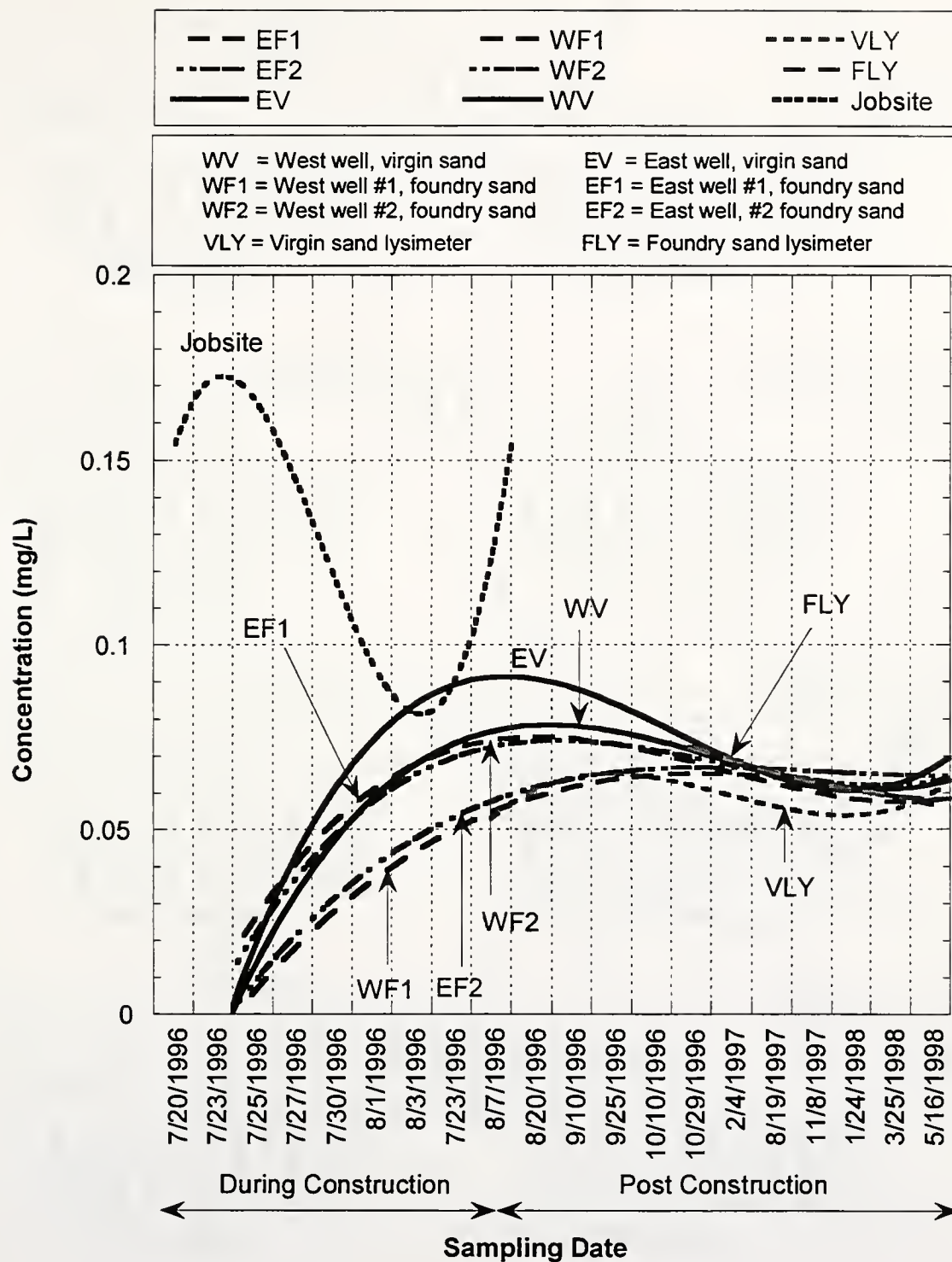


Figure 28 – Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Ni

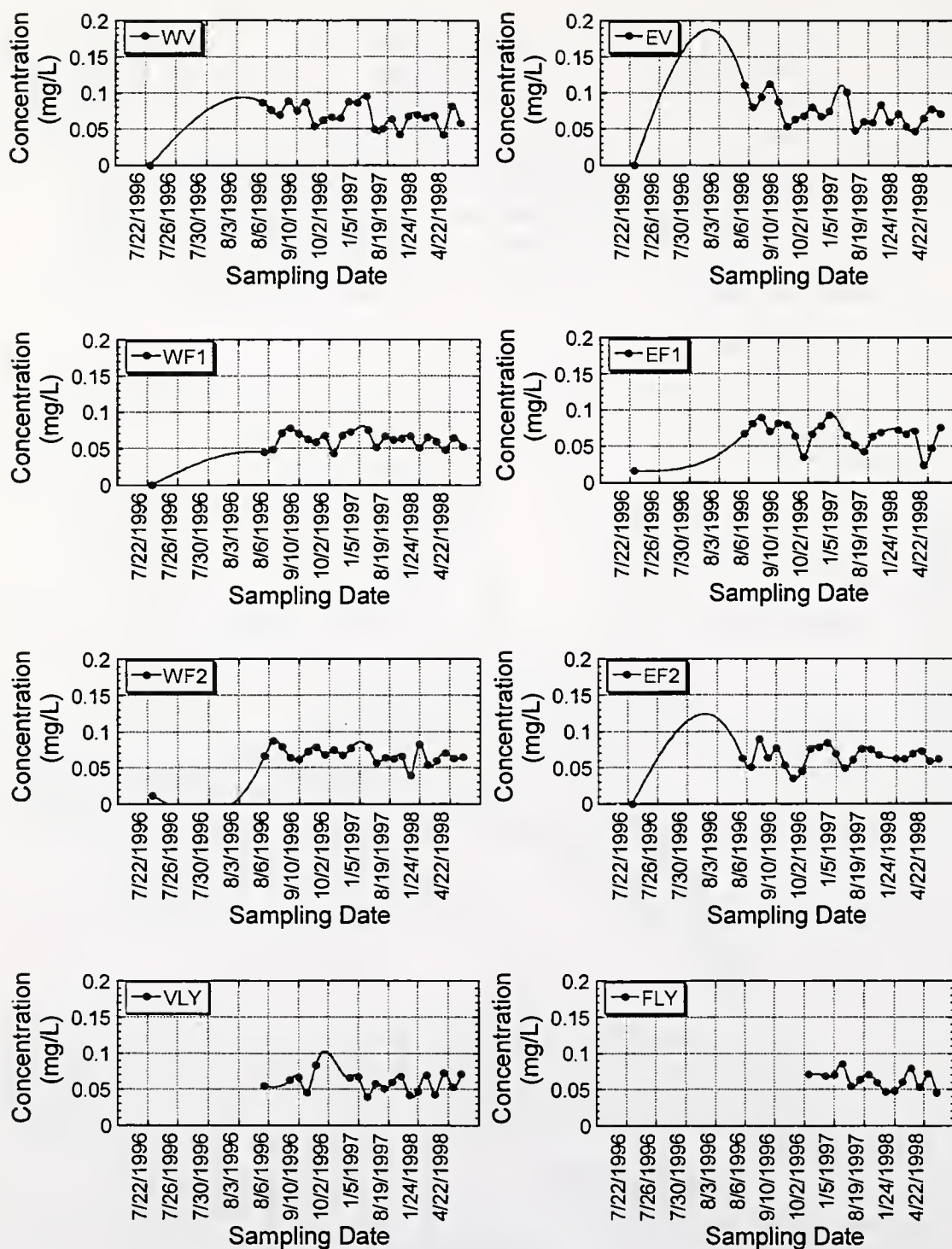


**Figure 29 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Ni**

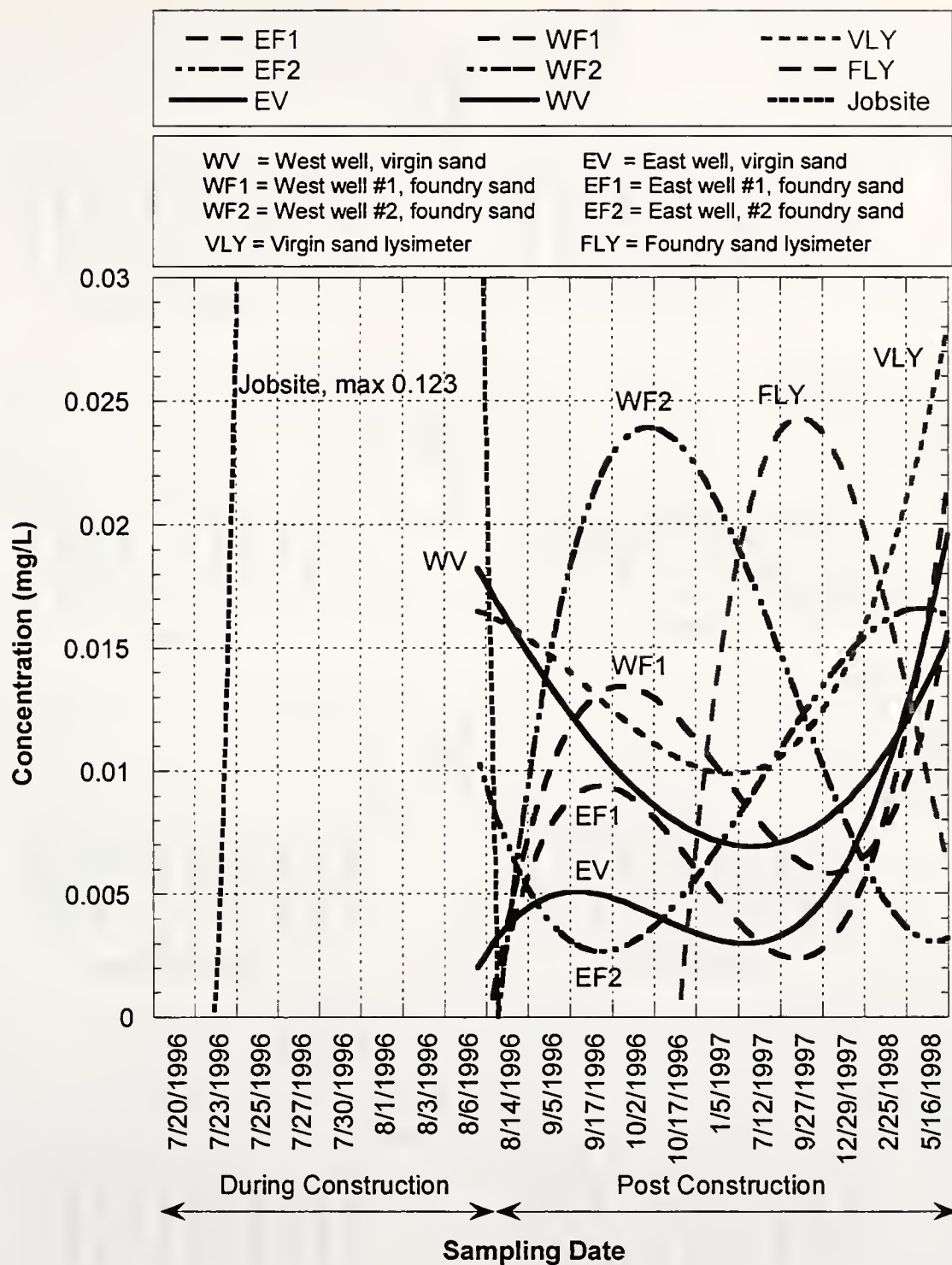


Figure 30 – Inductively Coupled Plasma (ICP) Spectrophotometer Testing for As

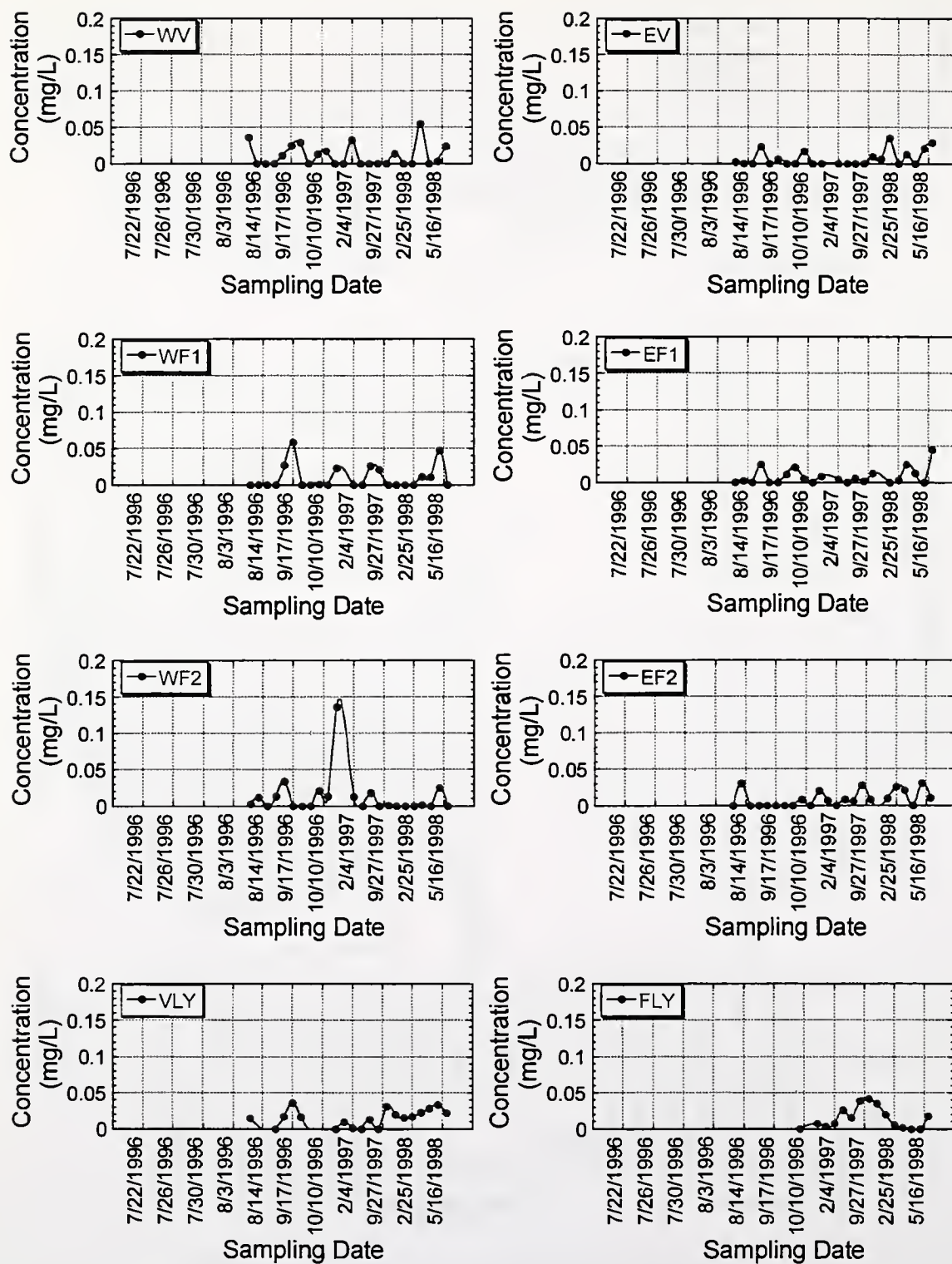


Figure 31 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for As

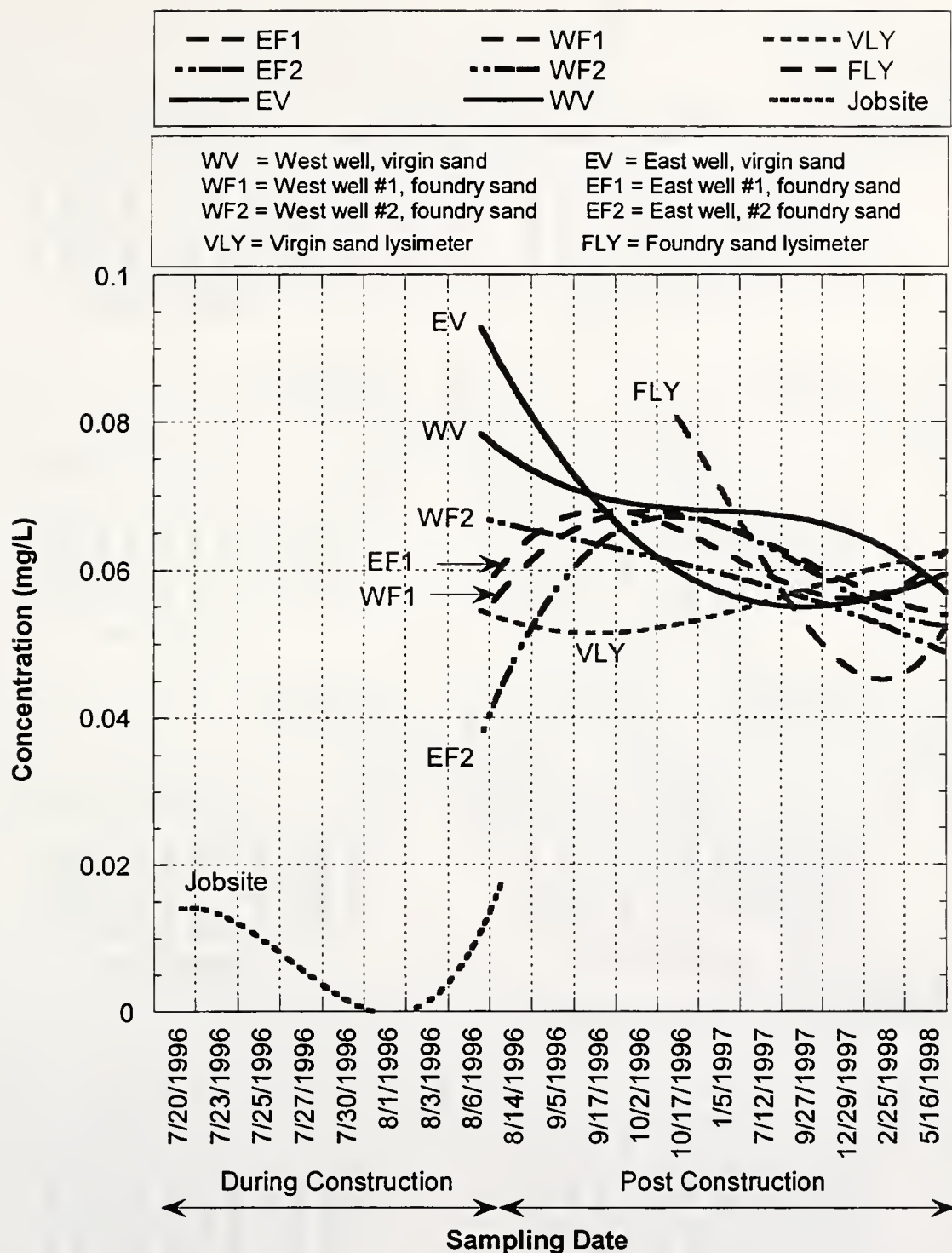


Figure 32 – Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Cr

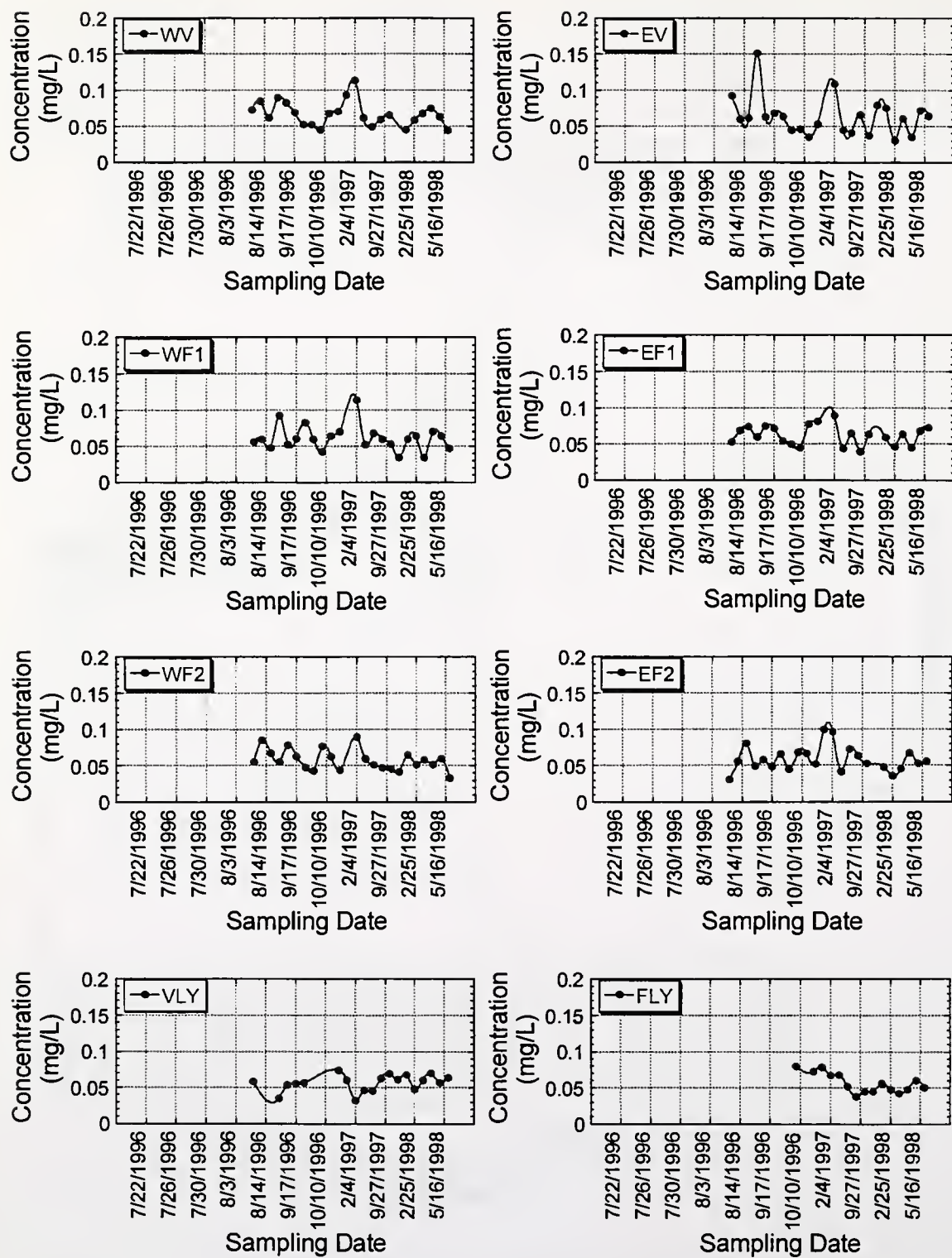


**Figure 33 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Cr**

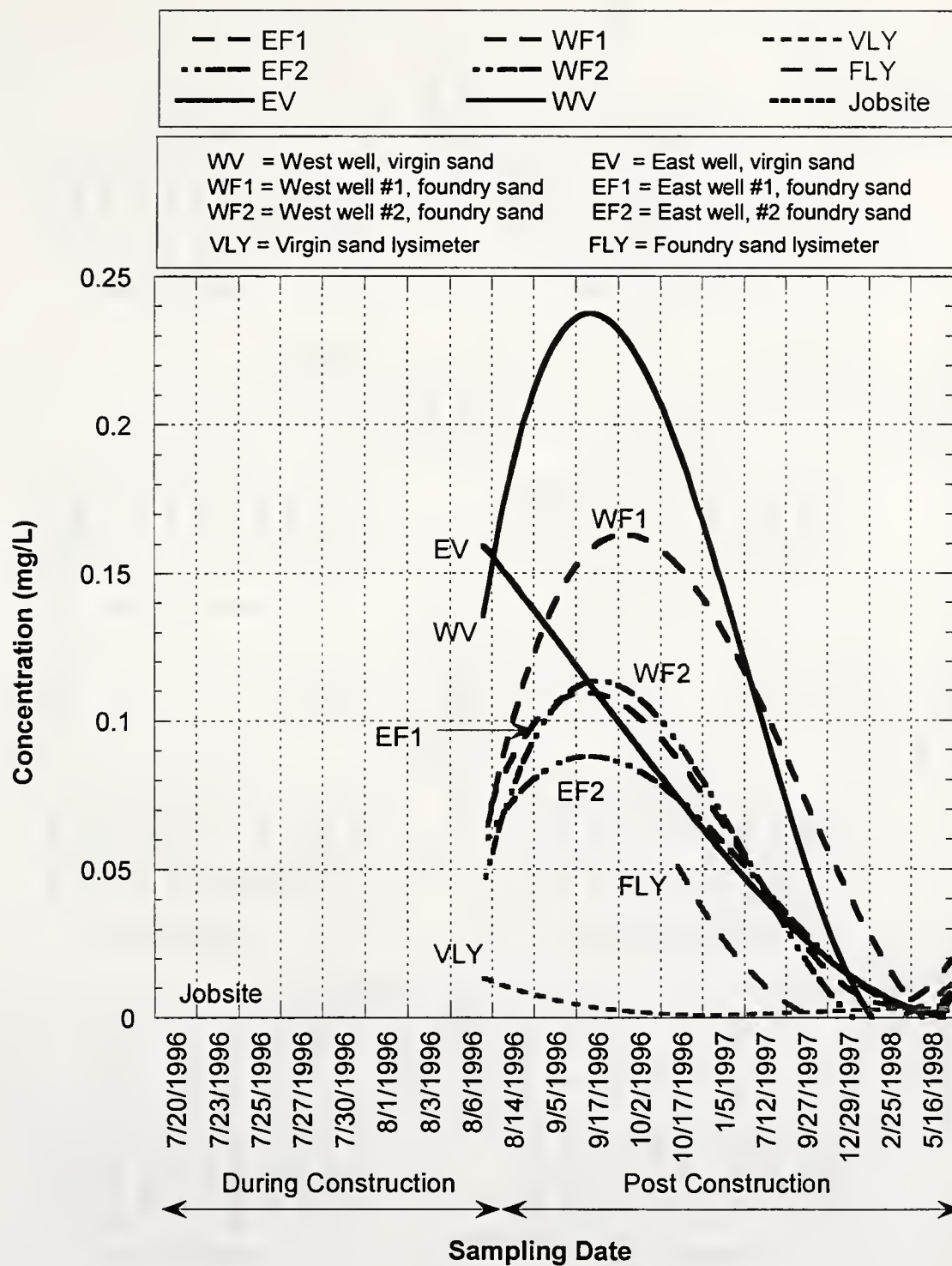


Figure 34 – Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Cu

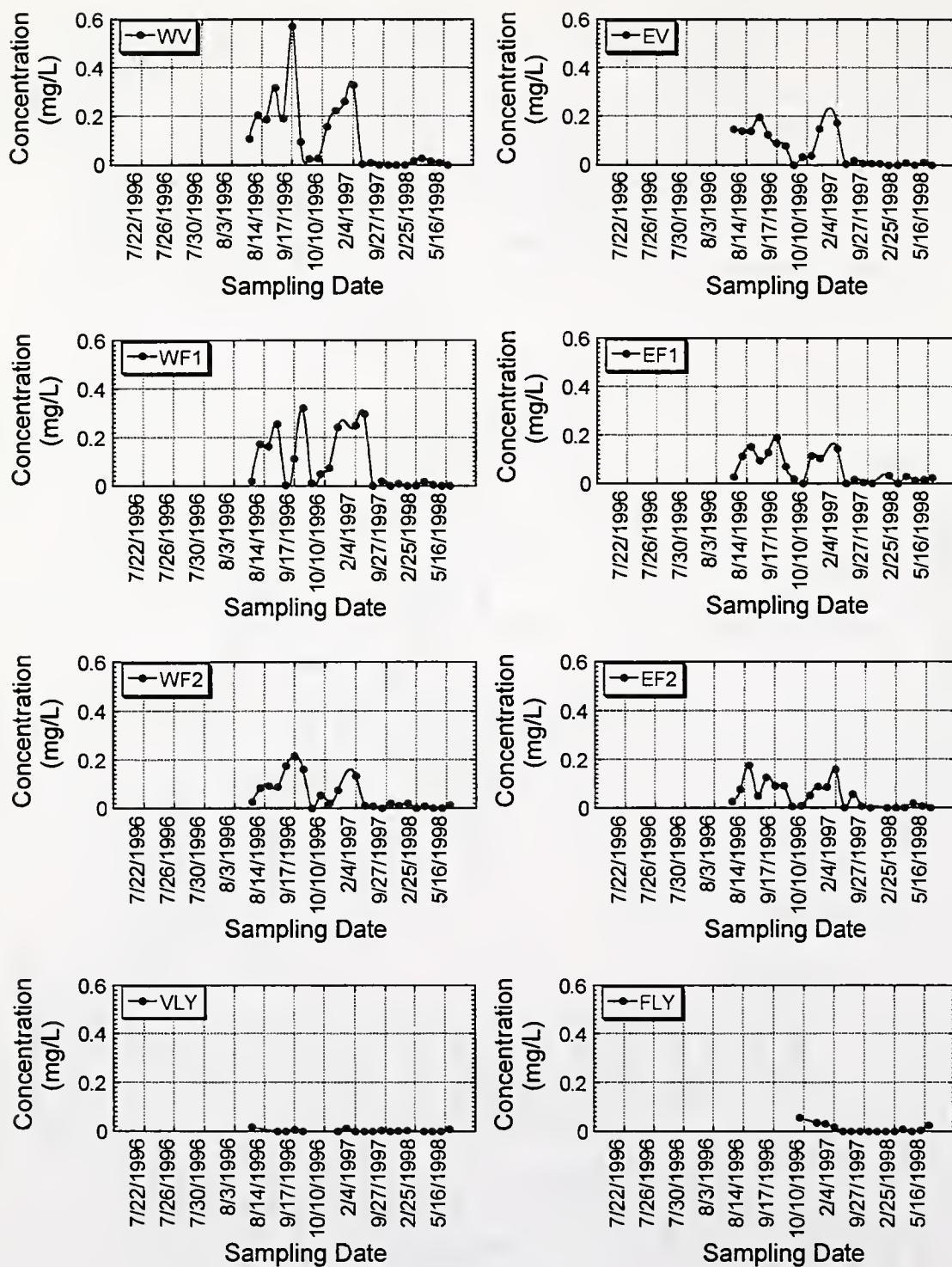


**Figure 35 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Cu**

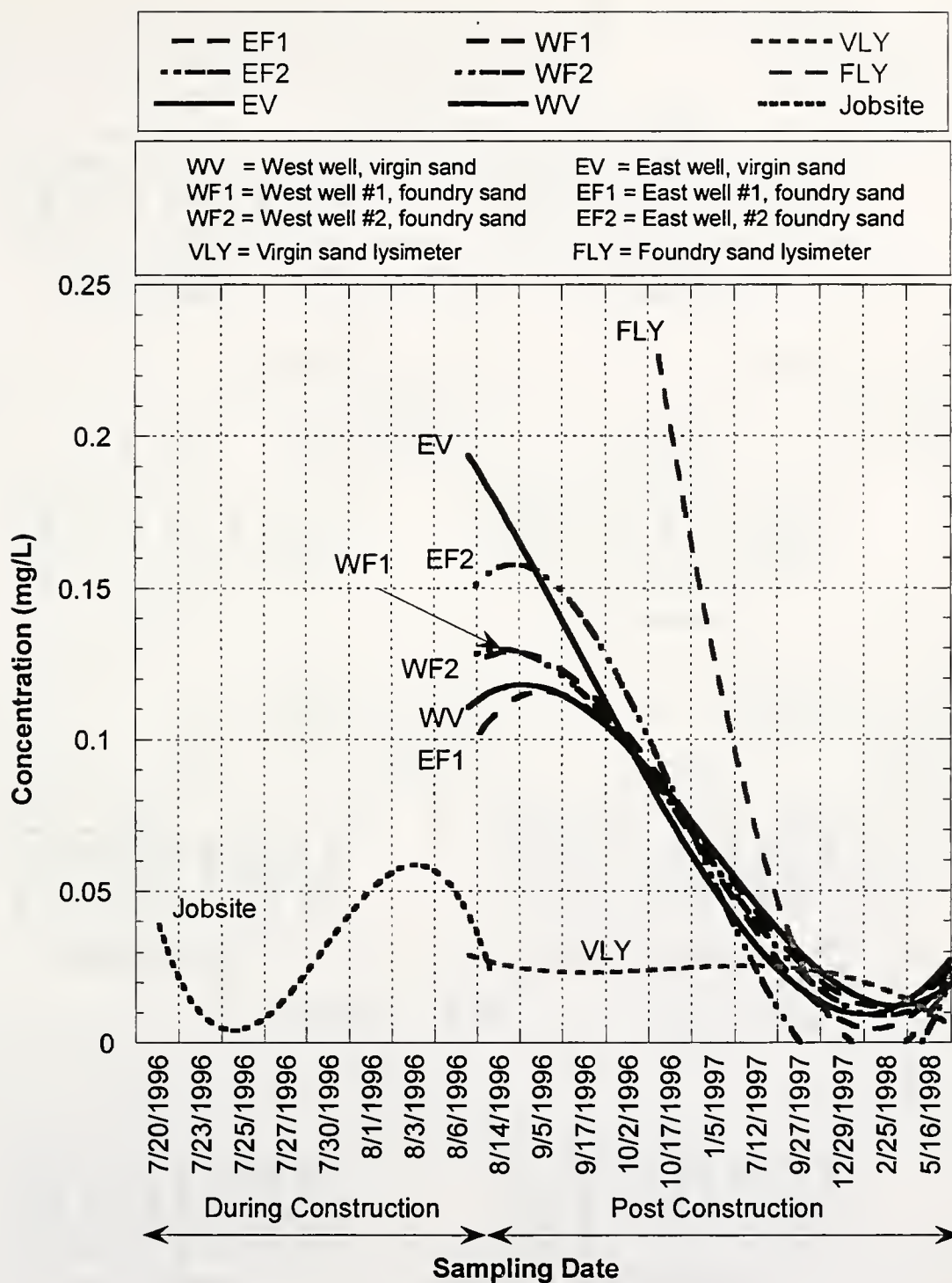


**Figure 36 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Pb**

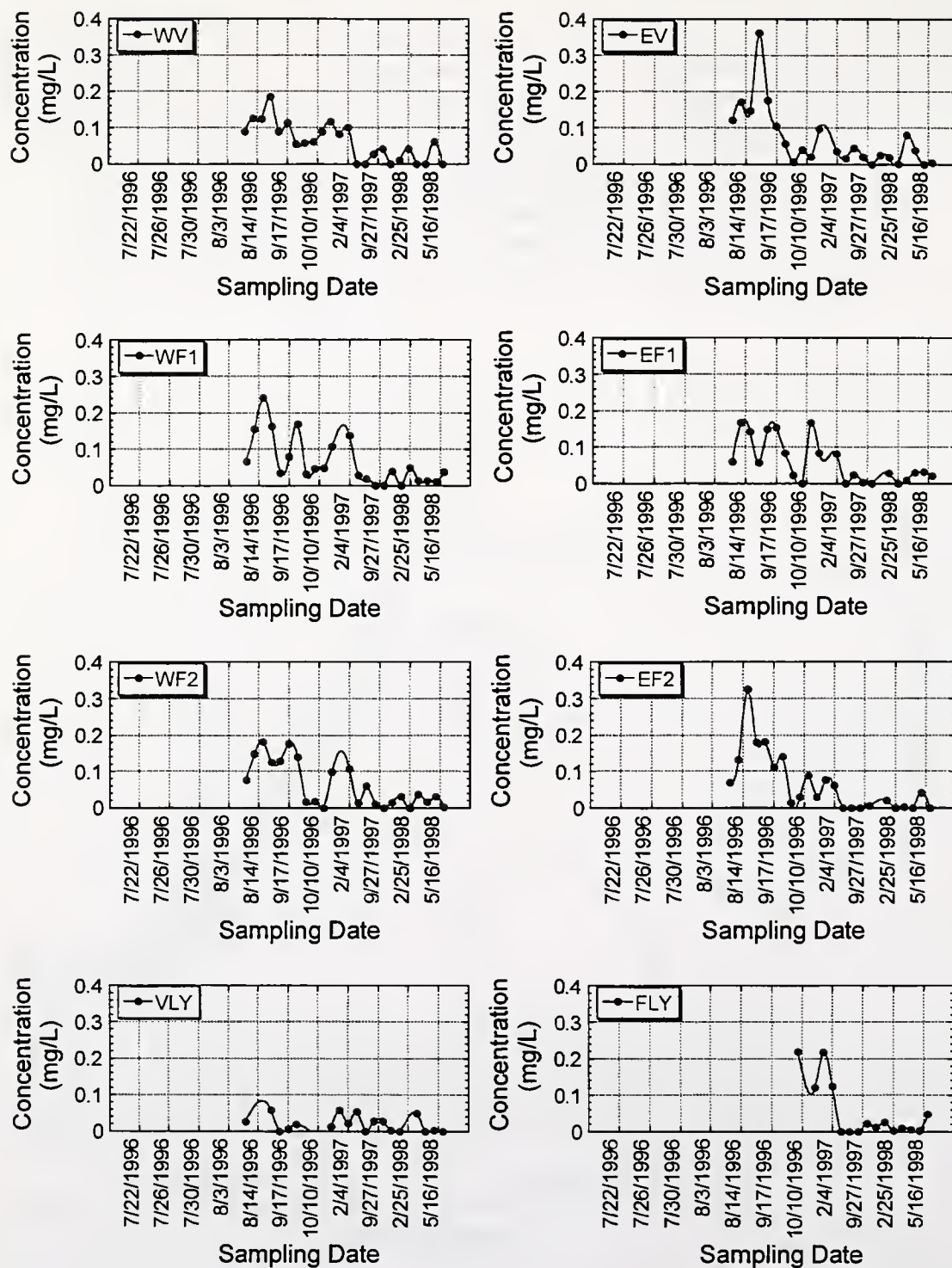


**Figure 37 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Pb**

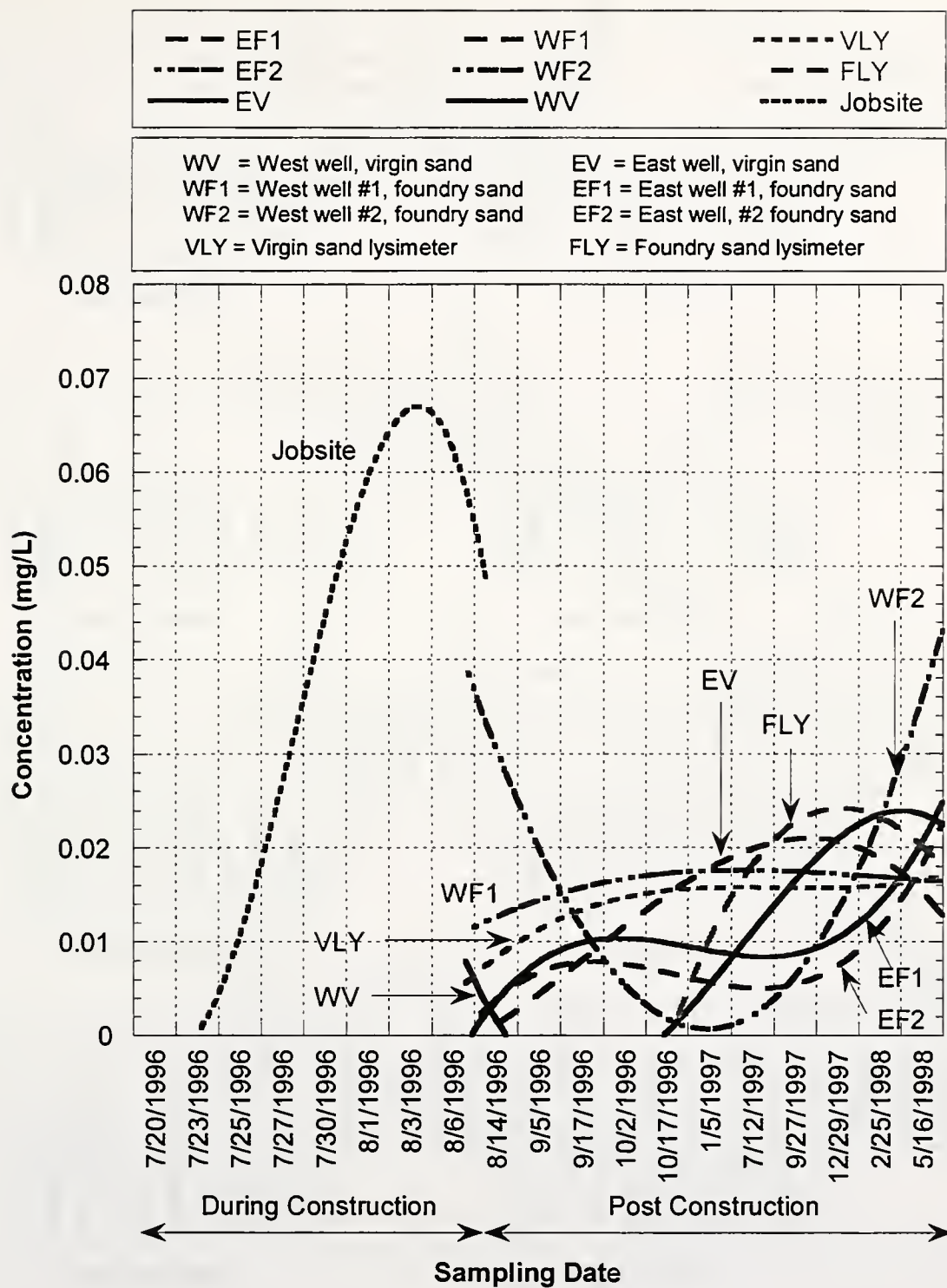


Figure 38 – Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Se

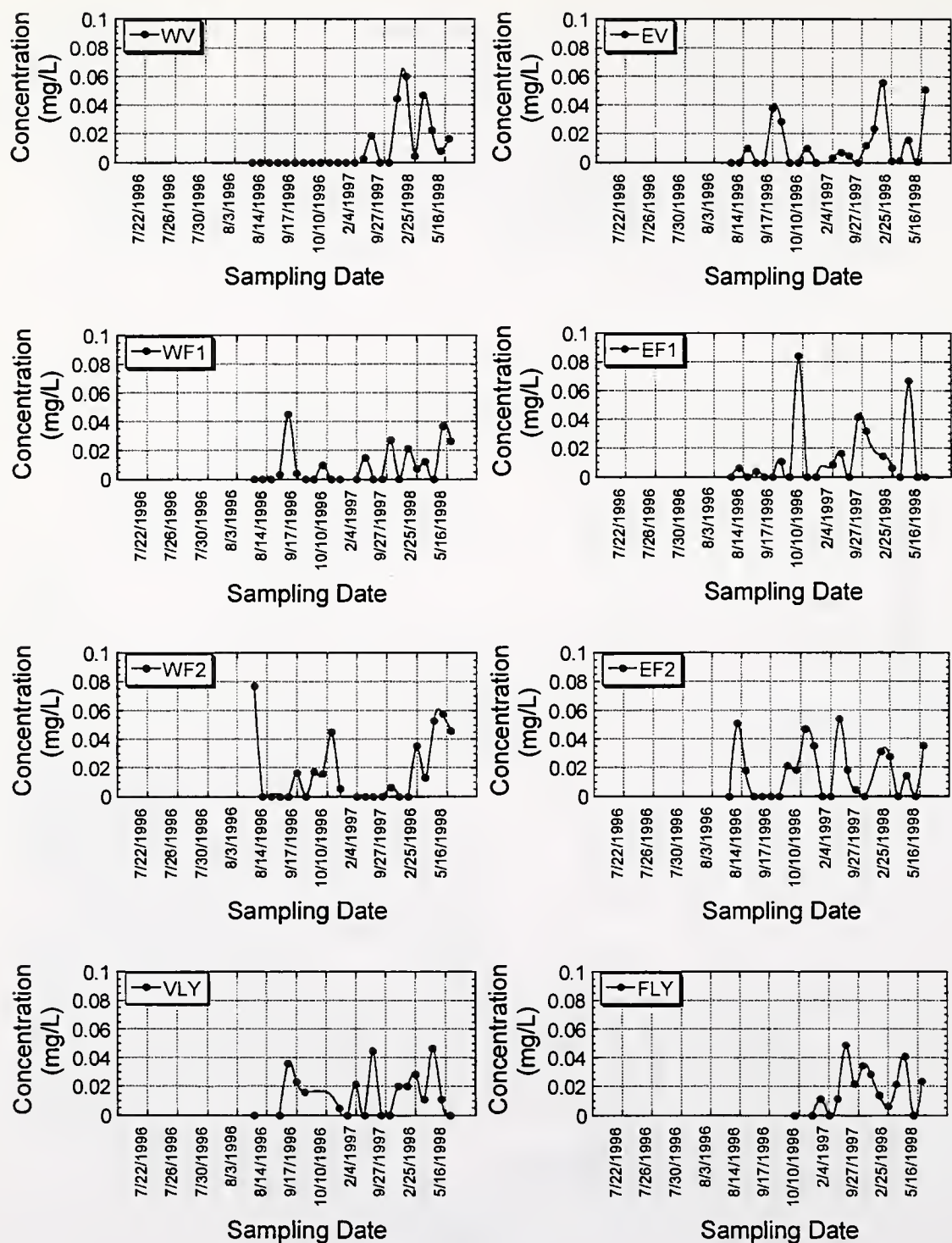


Figure 39 – Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Se

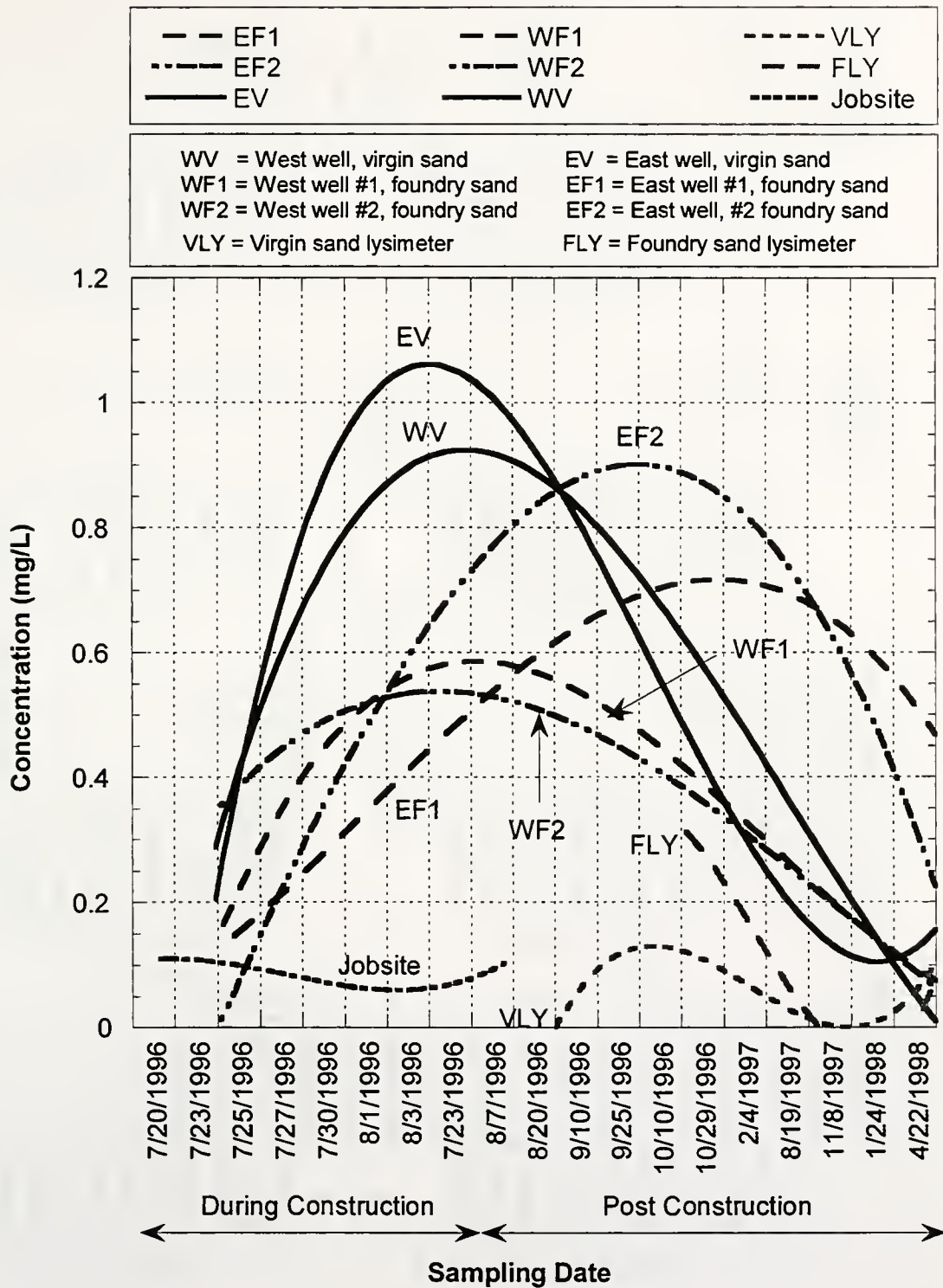


**Figure 40 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Zn**

Figure 41 – Inductively Coupled Plasma (ICP) Spectrophotometer Testing for Zn

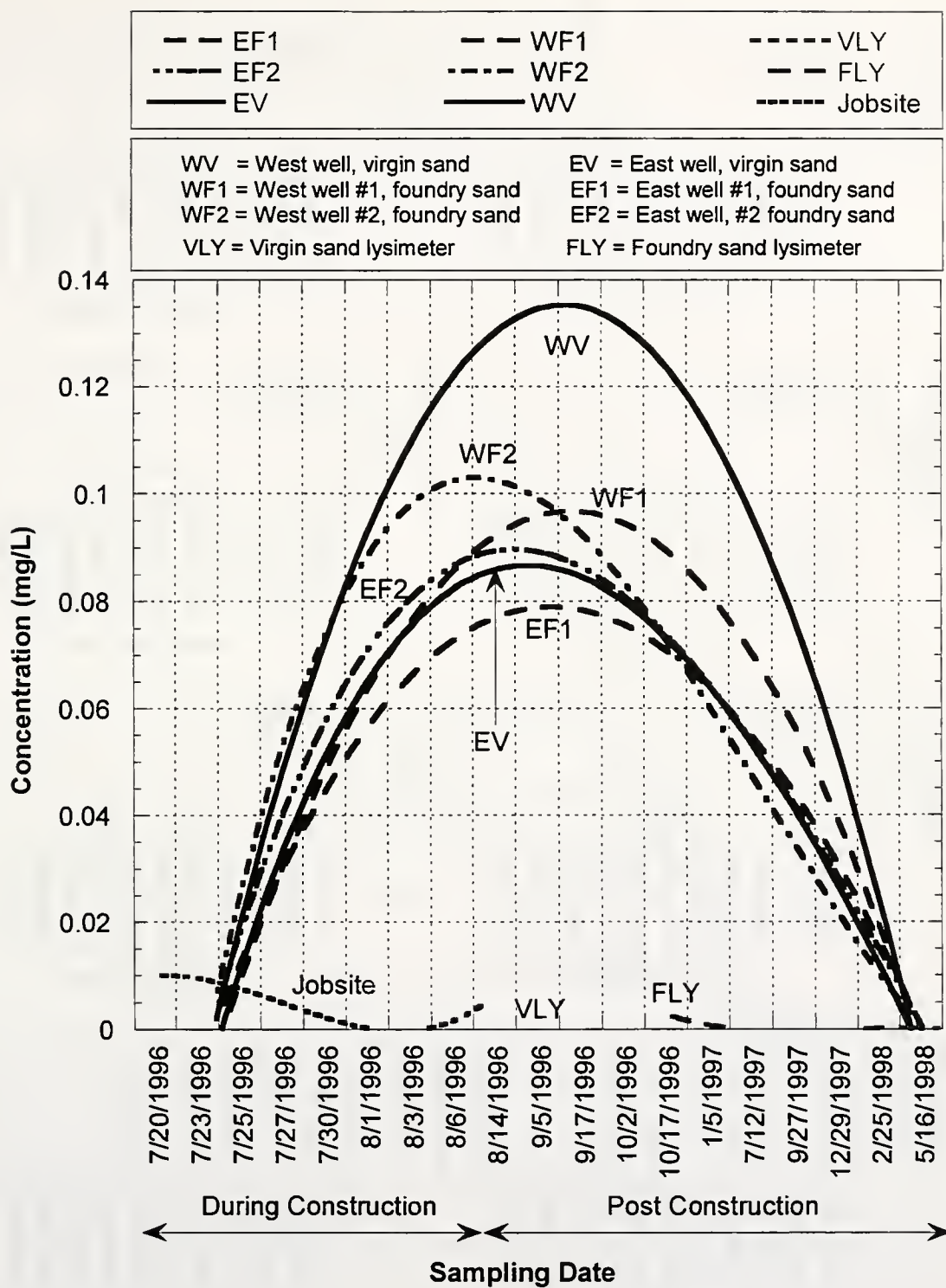


**Figure 42 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Cd**

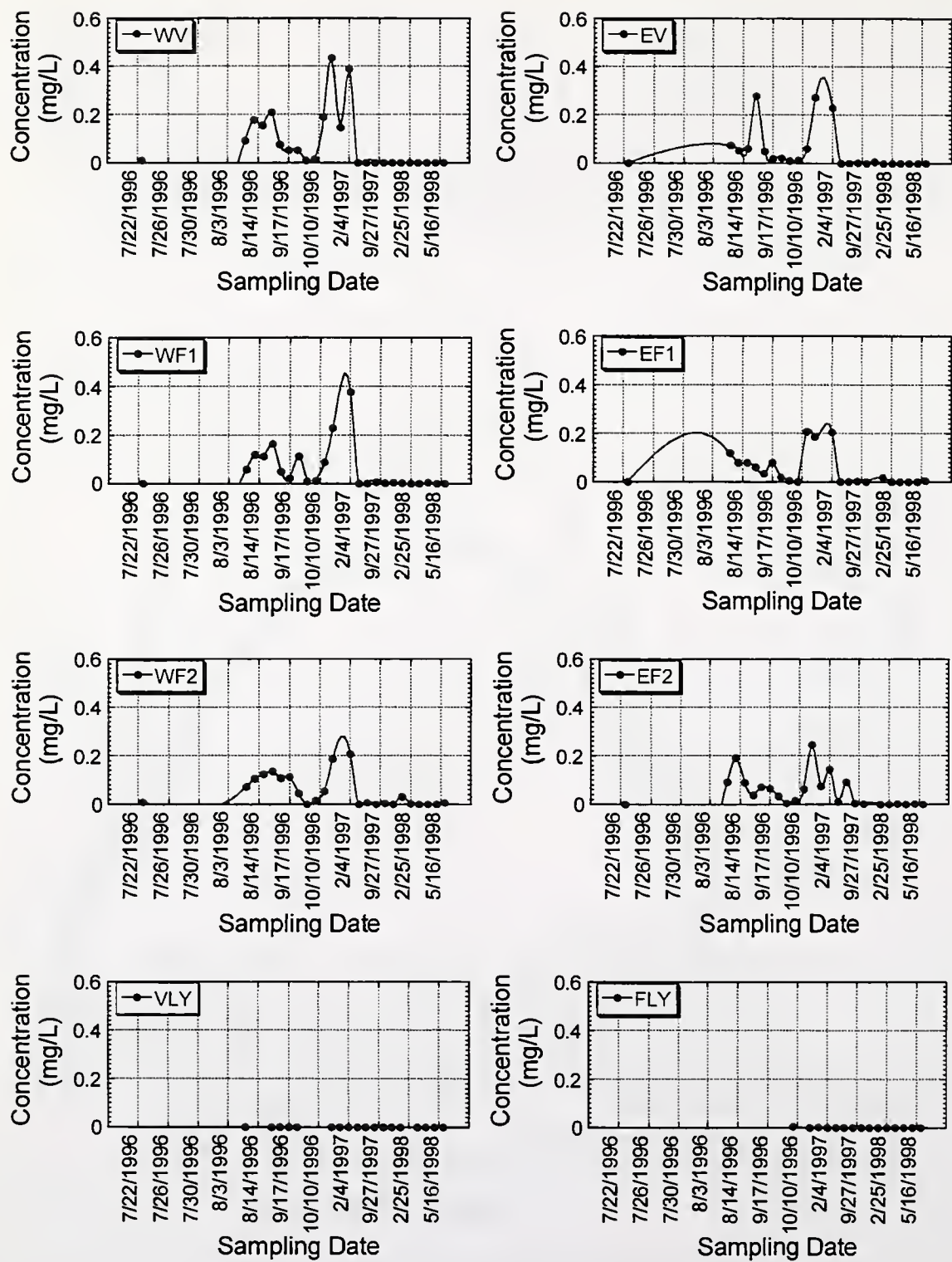


**Figure 43 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Cd**

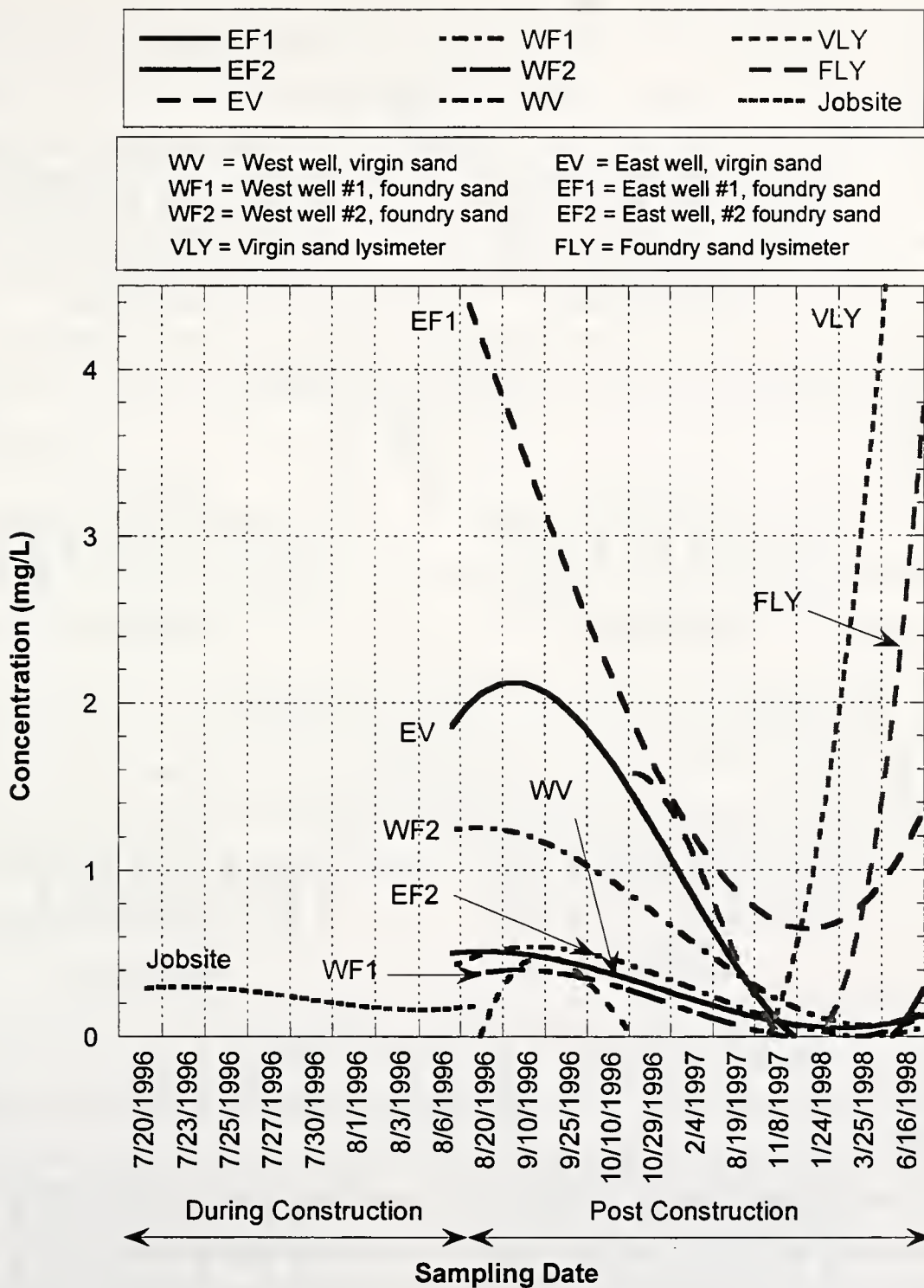


**Figure 44 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Mn**

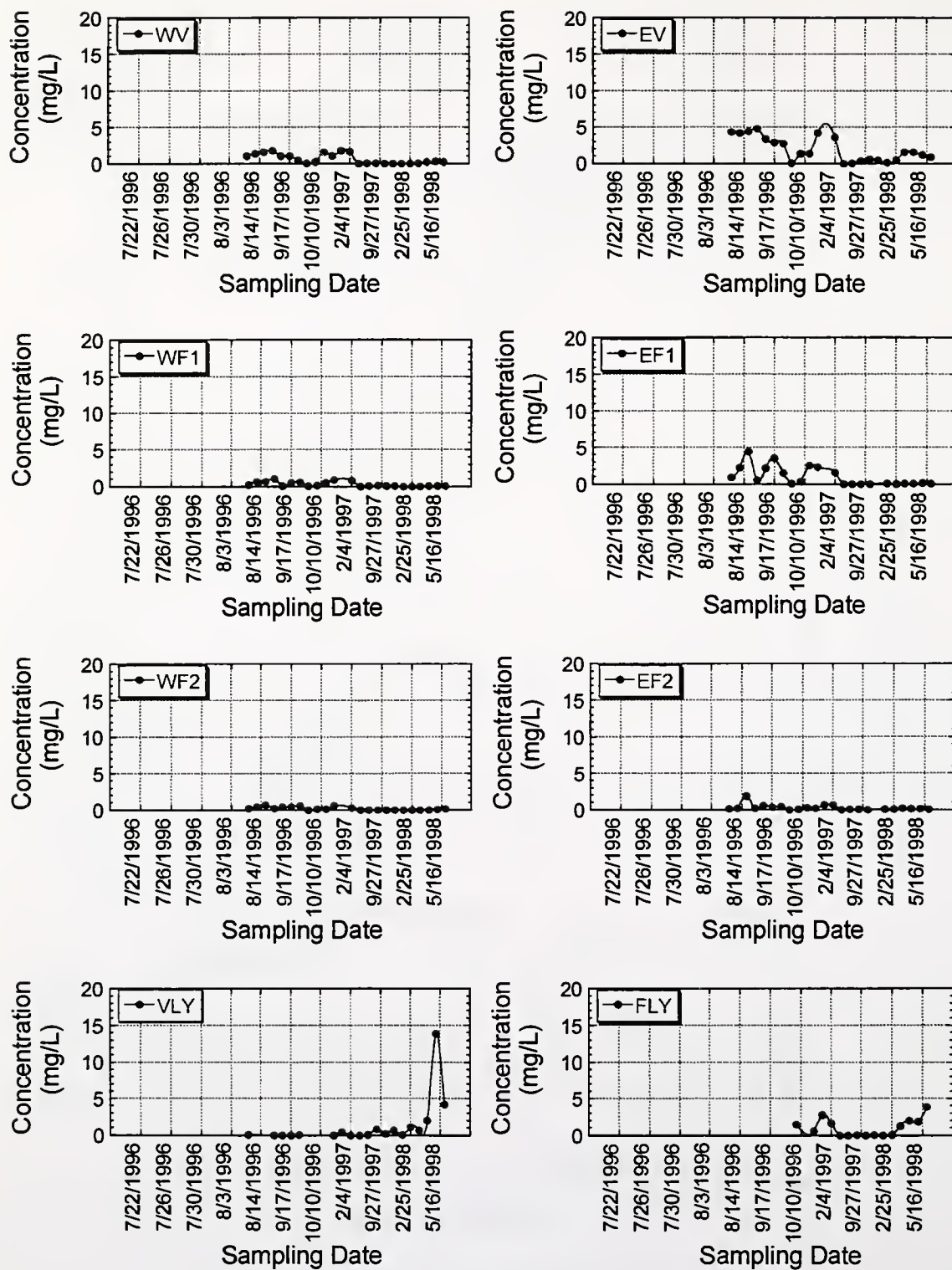


Figure 45 – Inductively Coupled Plasma (ICP)
Spectrophotometer Testing for Mn

conductivity of the WFS was considerably lower than that of the natural sand. In retrospect, a more appropriate control section would have been constructed using clay fill which typically would be specified by INDOT instead of B-borrow. The path of least resistance for surface waters after entering the WFS embankment, appears to have been laterally towards the WFS-natural sand interface and then down through the freely draining natural sand embankment and eventually manifesting in the EV groundwater well. While perhaps bearing little practical significance to field construction practices, this information would be useful in designing control sections for future WFS demonstration projects as well as providing insight into the trends of test results observed in this project.

Generally, a poor correlation (maximum $R^2=0.35$) was found between laboratory leachates from the WFS samples at the job site and post construction down-gradient well samples or lysimeters for any of the tests performed. This lack of correlation between laboratory leachate results and well and lysimeter leachate sample results agree with previous studies which indicated the difficulties encountered when trying to simulate complex field leaching conditions during laboratory characterization of these wastes (Boyle and Ham, 1979). More recent work (Blaha, 1986) found no conclusive method of correlating laboratory leachate concentrations with lysimeter or groundwater (down-gradient) contamination.

7.3 Economic Considerations of Waste Foundry Sand Reuse

As noted in Chapter 1, foundries can gain significant economic savings when WFS are used in beneficial reuse projects. This savings can be realized by extending the service lives of existing foundry-owned landfills and by avoiding or delaying stringent permitting requirements and costly permitting fees for siting new landfills. Additionally, generators are often willing to provide the WFS to a job site at no cost to the end user in lieu of incurring landfill tipping fees. Consequently, end users, such as contractors and DOTs, can obtain a free or very low-cost source of sand (fine aggregate). Transportation costs

for hauling the WFS, however, dictate the need for a relatively close proximity between the foundry and the job site.

For the CR 206 demonstration project, WFS were provided at no cost by the Auburn Foundry, resulting in no material costs to INDOT for the WFS embankment. However, Fox Contractors was paid \$0.61 per m^3 for compaction of the WFS. The B-borrow cost \$8.27 per m^3 , including compaction. This cost is unusually low as a result of an abundance of B-borrow available from a local borrow pit. On average, B-borrow would cost approximately \$13.08 per m^3 . The clay borrow used for the southern section of the project cost approximately \$3.92 per m^3 , including compaction, according to Mr. Ron Brown, INDOT Project Engineer.

Excluding the cost of trucking paid by the foundry, an economic comparison can be made between the WFS and the clay borrow as embankment fill. For this project, 42,815 m^3 of WFS cost \$26,117 dollars to compact. The same volume of clay borrow on this project would have cost \$167,835, including the costs of material, trucking, and compaction. Thus, the WFS embankment section cost \$141,718 less than a comparable clay embankment section, which translates to a savings of 84%. The extra cost of using B-borrow (in place of clay) for the north end of the embankment was \$150,993 (Mast, 1997).

The other expenditure which must be considered in an economic analysis was made by the foundry. The foundry reported spending approximately \$450,000 (\$10.52 per m^3) for trucking the WFS to the job site. This cost is considered high and should decrease as the foundry gains experience with hauling costs. Hauling costs can represent a significant economic investment on the part of the foundry, and such expenditures should be considered when planning a WFS reuse project. For the CR206 project, haul trucks drove 55 km round trip from the Auburn Foundry monofill to the construction site.

For the Auburn Foundry, the benefit of constructive reuse was primarily in terms of gained monofill space. The foundry regained approximately 1.25 years of service life in their monofill by donating the 42,815m³ of WFS to the project (Mast, 1997). The original cost of siting and constructing this monofill, combined with the current maintenance costs, translates into an estimated disposal cost of \$14.94 per m³ or for this project a savings to the foundry of \$639,656. According to Dan Hollenbeck, Environmental Engineer with the Auburn Foundry, the savings for a new landfill could be as much as \$1,406,852. This is due to the higher costs of siting and constructing a new landfill, due in part to stronger regulations for waste disposal. Subtracting the \$450,000 in trucking costs, the foundry's net savings (based on the current monofill cost) was \$189,656. Based on the cost of a new landfill, without adjustments for inflation, the net savings would be \$956,852. These calculations illustrate the potential savings available to foundries from the beneficial reuse of WFS. Table 11 summarizes the cost information.

7.4 INDOT Acceptance Criteria for Waste Foundry Sand Reuse Based on the Microtox™ Bioassay Test

As noted in Chapter 5, Bastian (1996) evaluated the ability of the Microtox™ bioassay test (90% Comparison Test) to assess the toxicity of WFS intended for constructive reuse. Sands from ferrous and non-ferrous foundries were tested in an effort to determine which sands would be suitable to be used as construction materials, by comparing Microtox™ responses of WFS with responses to virgin foundry and construction sands. This research reported the ability of the Microtox™ bioassay test to 'fingerprint' foundry residuals, determining which materials behaved similar to virgin sands and which materials exhibited inhibitory levels which would indicate a need for further chemical characterization prior to reuse or exclusion of the WFS.

Table 11: Summarized Costs and Savings Data for the CR 206 WFS Embankment Project

Contract Costs and Savings			
<u>WFS (42,815m³):</u>			
Material Cost of WFS*			\$0
Placement (Compaction) of WFS	\$0.61/m ³ x 42,815m ³		<u>\$26,117</u>
		Total Cost	\$26,117
*Transport cost paid by foundry			
<u>B-borrow (Natural Sand):</u>			
Material, Transport, and Compaction Cost of B-borrow in lieu of WFS (typically \$13.08/m ³)	\$8.27/m ³ x 42,815m ³		\$354,080
<u>Clay Borrow (locally available):</u>			
Material, Transport, and Compaction Cost of Clay Borrow in lieu of WFS	\$3.92/m ³ x 42,815m ³		\$167,835
<u>Contract Net Savings:</u>			
Using WFS in lieu of B-borrow:			\$327,963
Using WFS in lieu of Clay Borrow:			\$141,718
Auburn Foundry Savings			
Auburn Trucking Costs (55 km round trip)			\$450,000 (high)
Existing Monofill Service Life Extension, 1.25 yrs**			\$639,656
'New' Foundry Landfill Disposal Cost			\$1,406,852
Net Savings to Existing Monofill			\$189,656
Net Savings if 'New' Foundry Landfill			\$956,852
**Includes siting, construction, and maintenance costs			

The thirteen (13) virgin (natural) sand samples evaluated by Bastian consisted of 'clean' foundry sands and common construction sands from thirteen separate sources. Microtox™ responses to the virgin sands are shown in Figure 46 and indicate the sands are basically statistically equal based on ANOVA results of the raw data.

Using the virgin sand Microtox™ test results taken at the thirteen random locations the following required tests were performed for all the data. All observations were collected randomly and independently:

- A normality check was performed by location, time, control and sample. Each group (control and sample, for each time) contained five data points. The test results, using the Wilk-Shapiro Normality Test method indicated that the data was approximately normal.
- A homogeneity of variances test for the groups, each containing five observations, using the Barlett's Test of Equal Variance, indicated that the variances were statistically equal (homogeneous).

The next step was to compute the population normalized mean of 'acceptable' virgin sands (μ_A) and the corresponding population normalized standard deviation (σ). From these computations, the acceptance criteria was computed for use in accepting or rejecting a WFS source. This derivation is detailed in Appendix E, *INDOT Acceptance Criteria for Waste Foundry Sand Reuse Based on the Microtox™ Bioassay Test*.

7.5 Random Sampling of a Lot Within a Waste Foundry Sand Stockpile

As noted in Section 7.2.2, ANOVA of Microtox™ test results from the weathered stockpile and job site WFS leachates indicated both data sets were statistically equal for both the T5 (5 minute) and T15 (15 minute) tests, but were not statistically equal to each other. The WFS job site samples were believed to be more representative of the in-place embankment material as they were obtained over the entire depth of the monofill. INDOT recognized the need to develop a stratified sampling protocol using a statistically based sampling program for WFS stockpile characterization which addresses sample location and depth. In short, as WFS stockpiles can exhibit considerable variability, the end user must ensure sampling and testing is representative of the entire stockpile, for the

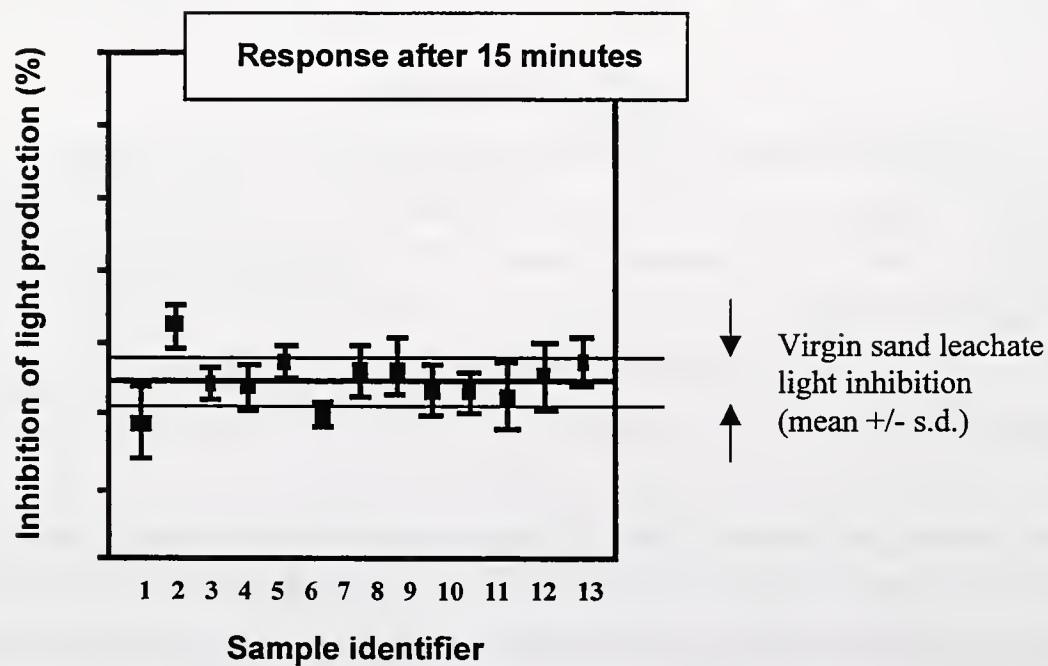
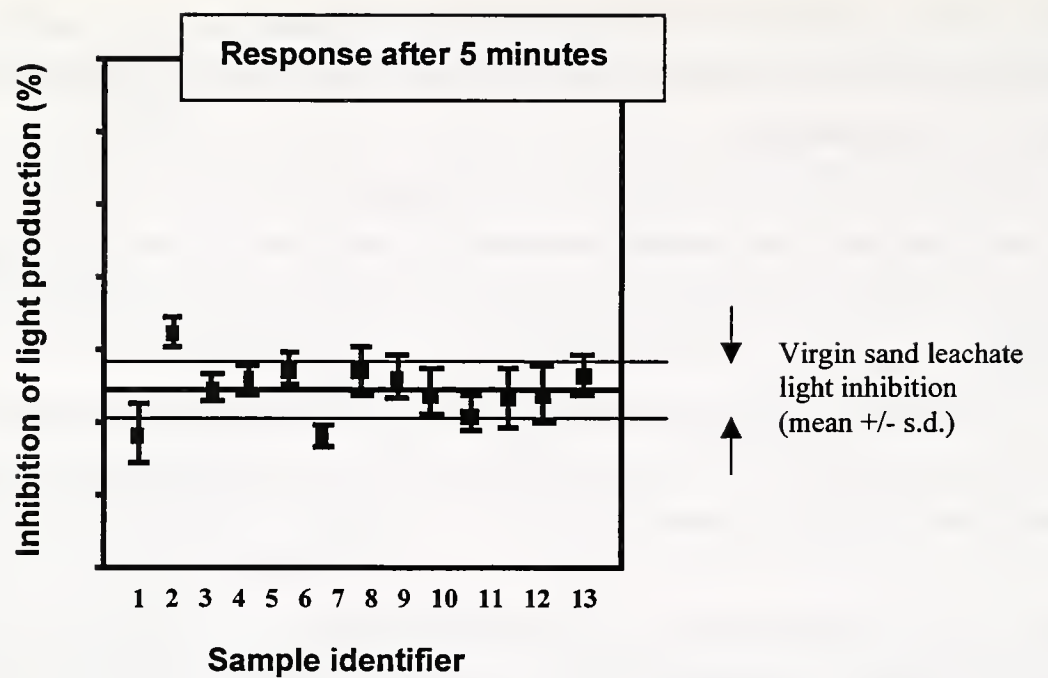


Figure 46 – MicrotoxTM Response to Virgin Sands
(Bastian, 1996)

intended reuse application. This sampling protocol is detailed in Appendix D, *Random Sampling Of A Lot Within A Waste Foundry Sand Stockpile*.

CHAPTER 8: CONCLUSIONS AND RECOMMENDATIONS

Conclusions

The following conclusions are reached as a result of geotechnical and environmental performance monitoring of a highway embankment constructed using waste foundry sand (WFS) from a ferrous metal casting foundry:

1. The geotechnical performance of the WFS section was comparable to that of the natural sand section, with small internal deformations and a high standard penetration resistance.
2. Dry unit weights of WFS were in general agreement with standard and modified Proctor compaction curves obtained using the ASTM Method B procedure. The Speedy Moisture Meter was preferred for the measurement of moisture contents in the field. Dry unit weights measured using a nuclear density gage were in good agreement with those obtained using the sand cone method.
3. Laboratory and field tests indicated the hydraulic conductivity for compacted WFS ranged from 1×10^{-8} to 7×10^{-7} m/s. In this study, compacted WFS was not considered a freely draining material.
4. WFS dust was controlled during construction by frequent watering of the working surface. Possible damage to construction equipment due to foreign objects in the WFS was a minor problem for this project. However, damage was minimal once the contractor became familiar with the material.
5. Differences in Microtox™ test results between weathered stockpile samples and job site 'grab' samples illustrate the importance of adequate characterization of a stockpile, for the intended use, with a statistically based representative sampling and testing program, prior to use.

6. Microtox™ bioassay test results indicate the WFS did not leach contaminants at inhibitions (toxicities) higher than those expected from common natural sands. Bioassay results indicate the WFS will not likely result in a negative environmental impact to the site. 'Toxic' responses from the foundry sand lysimeter (FLY) samples over a two week period in October 1996 are believed to be the result of leaching an inhibitory contaminant from the underlying geomembrane and not from the WFS. Attempts to correlate Microtox™ and Nitrotox inhibition results were unsuccessful.
7. Ion chromatography (IC) test results indicate F^- and $SO_4^{=}$ ions are migrating from the WFS, further validating the bioassay test results, but at concentrations below regulatory reuse criteria. Cl^- concentrations are below regulatory reuse criteria and likely not a result of leaching from the WFS. High nitrate (as N) concentrations were attributed to farm field runoff rather than leaching of the WFS. IC results indicate the WFS will not likely result in a negative environmental impact to the site.
8. Inductively coupled plasma (ICP) spectrophotometer testing for eleven metals indicated no well or lysimeter contamination from the WFS has occurred except for possibly Cd and Mn which exceeded IDEM Type III regulatory criteria. Cd and Mn well concentrations appear to be inherent to the groundwater quality as exceedences appeared in both up- and down-gradient wells and WFS job site leachate concentrations were below regulatory reuse criteria. Well and lysimeter metal concentrations for the remaining nine metals (Ag, As, Ba, Cr, Cu, Ni, Pb, Se, and Zn) were below regulatory reuse criteria and typically below 1996 drinking water standards (DWS).
9. Poor correlation was found between laboratory leachate results from WFS job site samples and field groundwater well and lysimeter leachate samples, indicating the difficulties encountered when trying to simulate complex field leaching conditions in the laboratory.
10. According to generators (foundries) of WFS, differences in state regulatory policies are the most frequently identified barriers to the reuse of WFS.

Regulatory policies and guidelines typically do not include methodologies for considering life-cycle issues (e.g., cost-benefit, risk). Test standards generally do not consider the background concentrations of certain elements (e.g., metals) resulting in WFS being subjected to more stringent standards than natural soils.

11. Foundries need to make a commitment to marketing and quality control of their WFS to provide potential customers an incentive to reuse WFS. In some instances, removal of a particular component (e.g. baghouse dust) from the composite waste stream can significantly lower resulting contaminant concentrations.
12. Departments of Transportation (DOTs) indicate that liability was their primary concern when considering whether to reuse a waste material such as WFS. Although a WFS reuser may be exempt from liability under RCRA regulations, the reuser may not be exempted from Superfund liability provisions. Some reusers have required indemnification from the foundry as a prerequisite to using the foundry's WFS.
13. Using WFS as an embankment material on this project resulted in a savings to the Indiana DOT and to the foundry. As a result of this WFS embankment project, the foundry regained 1.25 years of service life in their monofill. The WFS was provided to the job site by the foundry at no cost to the DOT.

Recommendations

1. Standard material tests can be used to determine the geotechnical properties of WFS.
2. Field compaction of WFS is probably best accomplished using heavy equipment which imparts a kneading action to the surface. Smooth drum and smooth drum vibratory rollers were found to be relatively ineffective in compacting WFS.
3. Dust should be controlled during construction by frequent watering of the compaction surface. Appropriate personal protection equipment (PPE) should also be addressed. Foundry operators who wish to reuse their WFS for construction should consider not incorporating foreign materials or bag house dust into their WFS stream.
4. Replicate testing of samples of known contaminant (organic and inorganic) concentrations, typical of WFS, would provide insight into whether the Microtox™ bioassay is more 'sensitive' than the Nitrotox bioassay.
5. A continued effort to test WFS from different foundries around Indiana is needed to provide information about other WFS sources suitable for reuse, as well as to verify the INDOT Acceptance Criteria. This information will help validate the use of bioassay testing as a quick and relatively inexpensive way to characterize WFS toxicity. In the interim, reusers should consider indemnification from the foundry when using their WFS, in particular when metal concentrations approximate the upper-limit of beneficial reuse criteria.

LIST OF REFERENCES

- Alleman, J.E. (1986). Respiration-Based Evaluation of Nitrification Inhibition Using Enriched *Nitrosomonas* Cultures, International Conference on Innovative Biological Treatment of Toxic Wastewaters, Scholze, R.J., Smith, E.D., Bandy, J.T., Yu, Y.C., and Basilico, J.V. (eds.), Noyes Data Corp., Park Ridge, NJ. pp. 643-651.
- Aprill, W., Sims, R.C., Sims, J.L., and Mathews, J.E. (1990). Assessing Detoxification and Degradation of Wood Preserving and Petroleum Wastes in Contaminated Soil, *Waste Management and Research*, 8, pp. 45-65.
- Azur Environmental (1997). The Microtox™ Comparison Test Detailed Protocol, Facsimile Transmittal, Carlsbad, California.
- Bambauer, R.A., Langer, H.J., and Yunovich, Y.M. (1993). New Binder Systems Befriends Environment, *Foundry Management and Technology*, pp. 20-24.
- Bastian, K.C. and Alleman, J.E., (1996). Environmental Bioassay Evaluation of Foundry Waste Residuals. Joint Highway Research Project, Final Report, FHWA/IN/JHRP-96-4, Purdue University, West Lafayette, IN.
- Bhat, S.T., Lovell, C.W., (1996). Use of Coal Combustion Residues and Waste Foundry Sands in Flowable Fill. Joint Highway Research Project, Final Report, FHWA/IN/JHRP-96/2, Purdue University, West Lafayette, IN.
- Bitton, G., and Koopman, B. (1986). Biochemical Tests for Toxicity Screening *Toxicity Testing Using Microorganisms*, Vol. 1, CRC Press, Boca Raton, FL, pp.27-55.
- Blaha, F.J., Ham, R.K., Boyle, W.C., Kunes, T.J., Nichols, D.G., and Stanforth, R.R. (1986). Leachate and Groundwater Quality in and around Ferrous Foundry Landfills and Comparisons to Leach Test Results, *Transaction of the American Foundrymen's Society*, 94, pp. 935-942.
- Blaise, C. (1991). Microbiotests in Aquatic Ecotoxicology: Characteristics, Utility, and Prospects, *Environmental Toxicology and Water Quality*, 6, pp. 145-155.

Blum, D.J.W. and Speece, R.E. (1991). Quantitative Structure-Activity Relationships for Chemical Toxicity to Environmental Bacteria, *Ecotoxicology and Environmental Safety*, 22, pp. 198-224.

Boane, T. (1997). Personal Communication, Azur Environmental, Carlsbad, CA.

Boyle, W.C. and Ham, R.K. (1979). Assessment of Leaching Potential From Foundry Process Solid Wastes, *Proceedings of the 34th Purdue Industrial Waste Conference*, West Lafayette, IN, pp. 129-147.

Bulich, A.A., Greene, M.W., and Isenberg, D.L. (1979). The Reliability of One Bacterial Luminescence Assay for the Determination of Toxicity of Pure Compounds and Complex Effluents, Unpublished Proceedings, American Society for Testing and Materials, Chicago, IL.

Bulich, A.A. (1984). MicrotoxTM—A Bacterial Toxicity Test with Several Environmental Applications, Toxicity Screening Procedures Using Bacterial Systems, D. Liu and B.J. Dutka (Eds.), pp. 55-64.

Bulich, A.A. (1986). Bioluminescence Assays, *Toxicity Testing Using Microorganisms*. Vol. 1, CRC Press, Boca Raton, FL, pp.57-74.

Casarini, D.C.P., Cunha, R.C.A., Sata, M.I.Z., and Sanchez, P.S. (1991). Evaluation of Toxicity Test Procedure to Define Loading Rates in a Land Treatment System, *Water Science and Technology* 24 (12), pp. 183-188.

Case, D.R. (1995). Resource Conservation and Recovery Act: Identification of Hazardous Wastes, *Environmental Law Handbook*, Sullivan, T.F.P. (ed.), Government Industries, Inc., Rockville, MD, pp. 48-54.

Casting Industry Suppliers Association, Sand Committee Task Force (1995). The Right Thing To Do – With Spent Sand, *Foundry Management and Technology* pp. 27-29.

Clegg, A.J. (1991). *Precision Casting Processes*, Pergamon Press, New York, NY.

Coleman, R.N., and Qureshi, A.A. (1985). MicrotoxTM and *Spirillum Volutans* Tests for Assessing Toxicity of Environmental Samples, *Journal of Environmental Contamination and Toxicology* 35, pp. 443-451.

Cullinane, M.J., and Channell, M. (1996). Evaluation of Solidification/Stabilization for Treating Explosives Contaminated Soils, *American Society for Testing and Materials*, 3rd Volume, ASTM STP 1240, Philadelphia, PA.

Dutka, B.J., and Kwan, K.K. (1982). Application of Four Bacterial Screening Procedures to Assess Changes in the Toxicity of Chemicals in Mixture, *Environmental Pollution (Series A)* 29, pp. 125-134.

Dutka, B.J. and Kwan, K.K. (1988). Battery of Screening Tests Approach Applied to Sediment Extracts, *Toxicity Assessment* 3, pp. 303-314.

Fister, B. (1997). Personal Communication, DOT, Wisconsin.

Foundry Management & Technology (1993a). Sand, Binders, Sand Preparation, and Coremaking, *Foundry Management & Technology*, pp. D3-D12.

Foundry Management and Technology (FM&T) (1993b). Sand Reclamation Planning Checklist, *Foundry Management and Technology*, pp. D20-D21.

Fox, P.J., and Mast, D.G. (1998). Geotechnical Performance of Highway Embankment Constructed Using Waste Foundry Sand, Joint Transportation Research Program, Final Report, FHWA/IN/JTRP-98-8, Purdue University, West Lafayette, IN.

Ham, R.K., Boyle, W.C. and Kunes, T.P. (1981). Leachability of Foundry Process Solid Wastes, ASCE, *Journal of the Environmental Engineering Division*, 107 (EE1), pp. 155-170.

Ham, R.K., Boyle, W.C., Engroff, E.C., and Fero, R.L. (1993a). Organic Compounds in Ferrous Foundry Process Waste Leachates, ASCE *Journal of the Environmental Engineering* 119 (1), pp. 34-55.

Ham, R.K., Boyle, W.C., Traeger, P., Wellender, D., Lovejoy, M., and Hippe, J. (1993b). Evaluation of Foundry Wastes for Use in Highway Construction, Final Report to Wisconsin Department of Natural Resources and Transportation.

Hollenbeck, D. (1997). Personal Communication, Auburn Foundry Inc., Auburn, IN.

Huber, B. (1994). Memorandum to Anthony S. Benton, Beneficial Reuses of Spent Ferrous Foundry Sands: INDOT's Exposure Under CERCLA and/or RCRA, Nov. 26, 1994, Indiana Department of Transportation, West Lafayette, IN.

Indiana Department of Transportation (INDOT) (1997). Acceptance Criteria for Waste Foundry Sand Reuse Based on the Microtox™ Bioassay Test, West Lafayette, IN.

Javed, S., (1993). Use of Waste Foundry Sand in Highway Construction, Unpublished Ph.D. Thesis, Purdue University, West Lafayette, IN.

Javed, S., and Lovell, C.W., (1994). Use of Waste Foundry Sand in Highway Construction, Joint Highway Research Project, Final Report, FHWA/TN/JHRP – 94/2J, Purdue University, West Lafayette, IN.

Javed, S., Lovell C.W., and Hollenbeck, D., (1994). Spent Foundry Sand and Its Uses in Civil Engineering , *Third International Conference on Environmental Issues and Waste Management in Energy and Mineral Production in Perth, Western Australia*, Brodie-Hall Research & Consultancy Centre Pty. Ltd. Western Australia: Curtin University of Technology, pp. 541-556.

Kaiser, K.L.E., and Palabrica, V.S. (1991). *Photobacterium Phosphoreum* Toxicity Data Index, *Water Pollution Research Journal of Canada*, 26 (3), pp. 361-431.

Kauffmann, P., Regan, R., Voigt, R. (1996). A Survey of State Environmental Regulations Impacting Beneficial Use of Foundry Residuals, Publication Submitted to American Foundryman's Society Transactions.

Krueger, R.C., Ham, R.K., and Boyle, W.C. (1989). The Variability of Ferrous Foundry Waste Leaching Characteristics and Comparison to Landfill Unsaturated Zone Leachate Quality, *43rd Purdue Industrial Waste Conference Proceedings*, pp. 605-616.

Kwan, K.K., Dutka, B.J., Rao, S.S., and Liu, D. (1990). Mutatox test: A New Test for Monitoring Environmental Genotoxic Agents, *Environmental Pollution* 65, pp. 323-332.

Levi, Y., Henriot, C., Coutant, J.P., Lucas, M., and Leger, G. (1989). Monitoring Acute Toxicity in Rivers With the Help of the Microtox™ Test, *Water Supply* 7, pp. 25-31.

Logue, C.L., Koopman, B., Brown, G.K., and Bitton, G. (1989). Toxicity Screening in a Large, Municipal Wastewater System, *Journal of the Water Pollution Control Federation* 61, pp. 632.

Lovejoy, M.A., Ham, R.K., Traeger, P.A., Wellander, D., Hippe, J., and Boyle, W.C. (1996). Evaluation of Selected Foundry Wastes for Use in Highway Construction, *Proceedings of the 19th International Madison Waste Conference*, Madison, Wisconsin, pp. 19-31.

Mast, D.G., (1977). Field Demonstration of a Highway Embankment Using Waste Foundry Sand, Unpublished M.S. Thesis, Purdue University, West Lafayette, IN.

McIntyre, S.W., Rundman, K.B., Baillod, C.R., Rush, P., Sandell, J., and Stillwell, B. (1992). Benefication and Reuse of Foundry Sand Residuals: A Preliminary Report, *Transactions of the American Foundrymen's Society*, 100, pp. 201-208.

Metcalf, M. (1997). Personal Communication, DOT, Illinois.

Microbics (1992). *Microtox Manual: Volume 4, Data Quality and Applying Results*, pp. 434-435.

Munkittrick, K.R., Power, E.A., and Sergy, G.A. (1991). The Relative Sensitivity of Microtox™, Daphnia, Rainbow Trout and Fathead Minnow Acute Lethality Tests, *Environmental Toxicology and Water Quality* 6, pp. 35-62.

Naik, T.R., Patel, V.M., Parikh, D.M., and Tharaniyil, M.P. (1994). "Utilization of Used Foundry Sand in Concrete," *Journal of Materials in Civil Engineering*, ASCE Volume 6, No. 2, May 1994, pp. 254-263.

Nealson, K.H. and Hastings, J.W. (1979). Bacterial Bioluminescence: Its Control and Ecological Significance, *Microbiology Review* 43 (4), pp. 496-518.

Partridge, B.K., Alleman, J.E., and Huber B.W., (1997). Perspectives on Liability for Constructive Reuse of High Volume Waste Streams, *Transportation Research Record 1577*, Transportation Research Board, National Research Council, Washington, D.C., pp. 109-117.

Pastorok, R.A. and Becker, D.S. (1990). Comparative Sensitivity of Sediment Toxicity Bioassays at Three Superfund Sites in Puget Sound, *Aquatic Toxicology and Risk Assessment: Thirteenth Volume*, ASTM STP 1096, W.G. Landis and W.H. Van der Schalie (Eds.), American Society for Testing and Materials, Philadelphia, PA, pp. 123-139.

Qureshi, A.A., Coleman, R.N., and Paran, J.H. (1984). Evaluation and Refinement of the Microtox™ Test for Use in Toxicity Screening, *Toxicity Screening Procedures Using Bacterial Systems*, D. Liu and B.J. Dutka (Eds.), National Water Research Institute, Ontario, Canada, pp. 1-22.

Qureshi, A.A., Flood, K.W., Thompson, S.R., Janhurst, S.M., Inniss, C.S., and Rokosh, D.A. (1982). Comparison of a Luminescent Bacterial Test with Other Bioassays for Determining Toxicity of Pure Compounds and Complex Effluents, *Aquatic Toxicology and Hazard Assessment: Fifth Conference*, ASTM STP 766, J.G. Pearson, R.B. Foster, and W.E. Bishop, (Eds.), Philadelphia, PA, pp. 179-195.

Regan, R., (1996). Personal Communication, State College, Pennsylvania.

Ribo, J.M. and Rogers, F. (1990). Toxicity of Mixtures of Aquatic Contaminants Using the Luminescent Bacteria Bioassay, *Toxicity Assessment* 5, pp. 135-152.

Salazar, A., (1997). Bacterial Bioassay Monitoring of a Highway Embankment Constructed Using Waste Foundry Sand, Unpublished M.S. Thesis, Purdue University, West Lafayette, IN.

Sanchez, P.S., Sato, M.I.Z., Paschoal, C.M.R.B., Alvez, M.N., Furlan, E.V., and Martins, M.T. (1988). Toxicity Assessment of Industrial Effluents from Sao Paulo State, Brazil, Using Short-Term Microbial Assays, *Toxicity Assessment* 3, pp. 55-80.

Schiewe, M.H., Hawk, E.G., Actor, D.I., and Krahn, M.M. (1985). Use of a Bacterial Bioluminescence Assay to Assess Toxicity of Contaminated Marine Sediments, *Canadian Journal of Fisheries and Aquatic Science* 42, pp. 1244-1248.

Serat, W.F., Budinger, F.E. Jr., and Mueller, P.K. (1965). Evaluation of Biological Effects of Air Pollutants by Use of Luminescent Bacteria, *Journal of Bacteriology* 90 (3), pp. 832-833.

Slattery, G.H. (1988). TRE—Toxics Reduction Evaluation. Case Study—Operations and Managements Viewpoint Lessons Learned, Unpublished, Patapaco WWTP, Baltimore, MD, WPCA Meeting, May 1-3.

Stern, K. (1995). The Use of Spent Foundry Sand in Flowable Fill in Ohio, *Foundry Management and Technology*, pp. 78-86.

Tung, K.K., Scheibner, G., Miller, T., and Bulich, A.A. (1990). A New Method for Testing Soil and Sediment Samples, Presented at the SETAC Conference, Nov. 1990.

Walborn, D. (1997). Personal Communication, Reuse Recovery Corporation of West Michigan, Michigan.

Walker, J.D. (1988). Relative Sensitivity of Algae, Bacteria, Invertebrates, and Fish to Phenol: Analysis of 234 Tests Conducted for More Than 149 Species, *Toxicity Assessment* 3, pp. 415-447.

Westervelt, W.W., (1988). A Toxicity Assessment of Foundry Residual Wastes, Unpublished M.S. Thesis, Pennsylvania State University, State College, PA.

Ziegler, M.J. (1994). Foundry Sand Basics, *Foundry Management and Technology*, pp. 25-29.

APPENDIX A
MICROTOX™ 90% COMPARISON TEST PROTOCOL

(Azur Environmental 1997)

Analyzer Preparation

1. Fill a 10 ml beaker with Microtox diluent
2. Fill a 10 beaker with sample, and add osmotic adjusting solution (OAS if necessary)
3. Place cuvettes in incubator rows B, C, D, E, REAGENT well, and F1
4. Using a 2.5 ml electronic pipette, add 1.0 ml reconstitution solution to the cuvette in the REAGENT well
5. Using the same pipette and tip, add 1.5 ml diluent to B1, B3, B5, D2, D4, and F1
6. Using the same pipette and tip, add 1.5 ml sample to B2, B4, D1, D3, and D5
7. Wait 5 minutes

Reagent Preparation

1. Reconstitute a vial of reagent by quickly adding the solution in the REAGENT well to the vial of freeze-dried bacteria, swirling the vial 3-4 times, and pouring the solution into the REAGENT well cuvette
2. Using a 1 ml pipette, mix the reagent 10 times with the pipette set to 0.5 ml
3. Using a 250 μ l pipette, transfer 150 μ l reagent to F1
4. Using the 1 ml pipette, mix the solution in F1 with the pipette set to 0.5 ml
5. Using a 100 μ l electronic pipette, transfer 100 μ l diluted reagent from F1 to the cuvettes in wells C1 through C5 and E1 through E5
6. Tap cuvettes on the machine a few times to assure even covering of the bottom of the cuvettes with liquid
7. Wait 15 minutes

While you wait.

Computer Preparation

1. Call up the comparison test program
2. Select "Start Testing"
3. Enter a file name
4. Enter one line of sample description
5. Set test parameters

Test Protocol

1. Place C1 cuvette in the READ well. Press the SET button and wait for the cuvette to be lowered and raised again and the green READY light to come back on
2. Touch the computer space bar key
3. Read initial light levels as prompted by the computer screen. Then immediately...
4. ...Using the 2.5 ml electronic pipette, make the following 0.9 ml transfers:

B1 to C1	D1 to E1
B2 to C2	D2 to E2
B3 to C3	D3 to E3

B4 to C4

D4 to E4

B5 to C5

D5 to E5

Touch the space bar when transfers have been completed.

5. When timer sounds, read light levels as prompted by the computer screen.

Data Reduction

1. Select Run Statistics from the Master Menu, select the file on which you wish to run statistics
2. Follow direction on the computer screen. When you get to the Calculations Menu, make a data report by selecting Print Report and printing on paper and to the hard disk

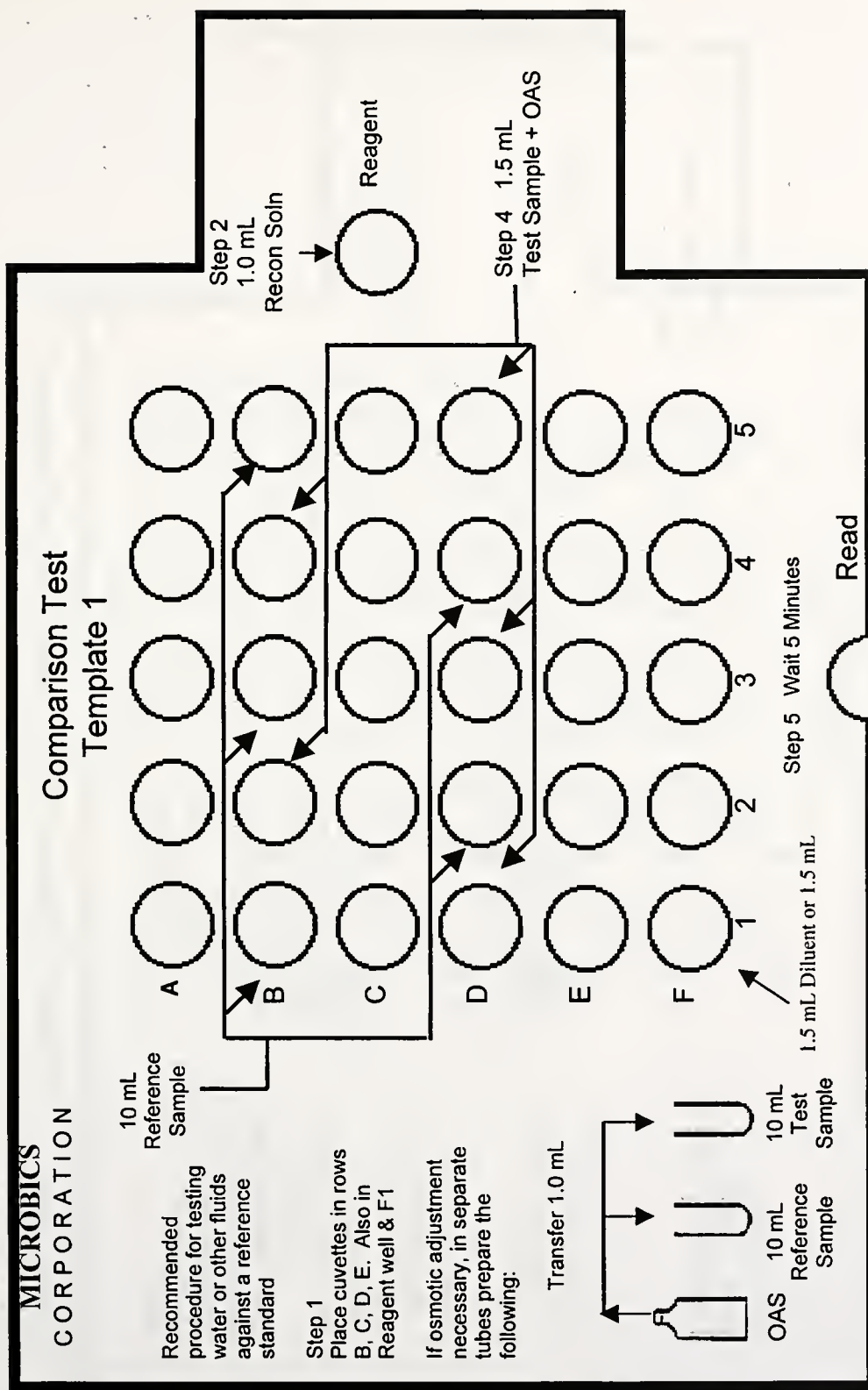


Figure 47 - Microtox™ 90% Comparison Test, Template 1

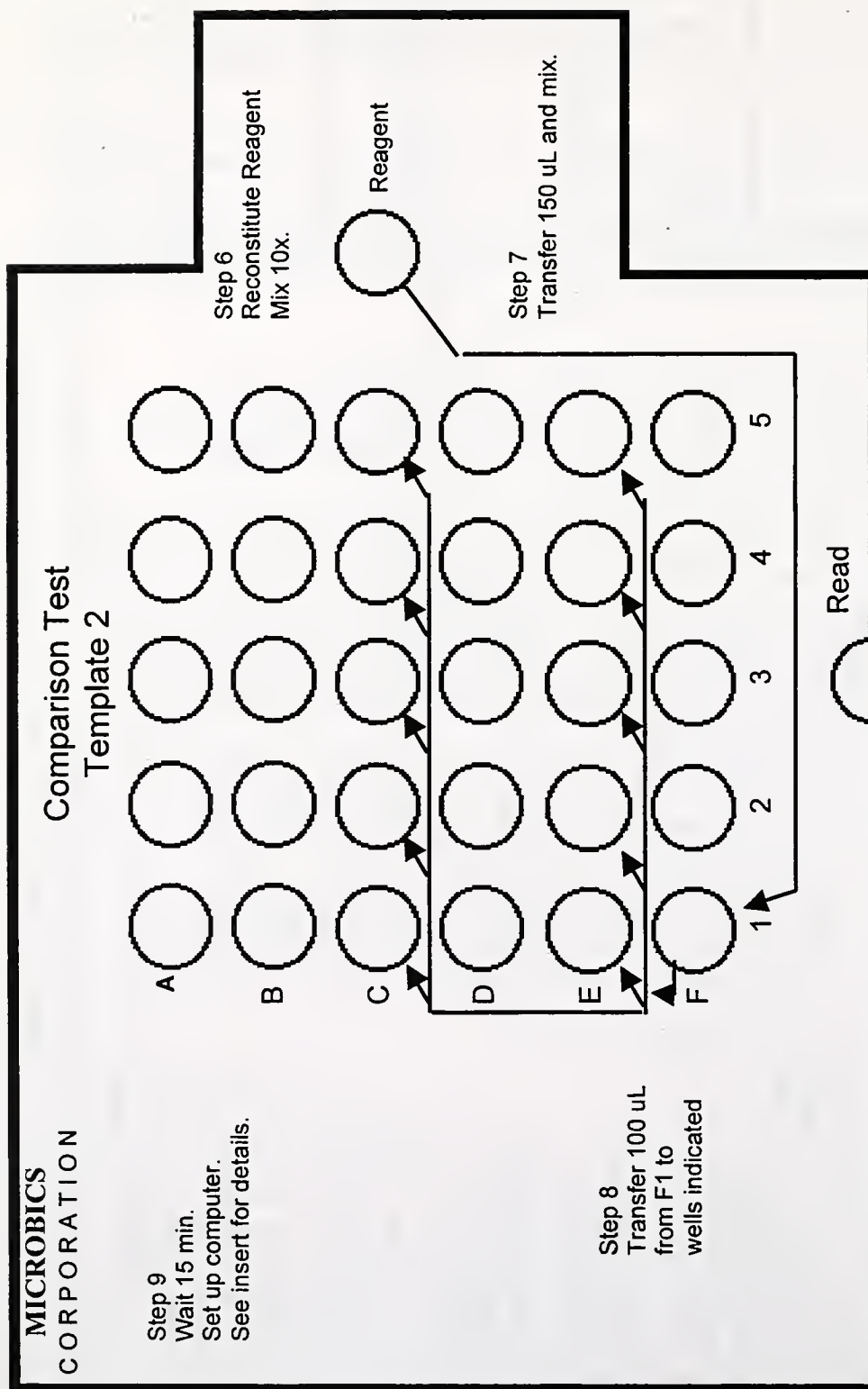


Figure 48 - Microtox™ 90% Comparison Test, Template 2

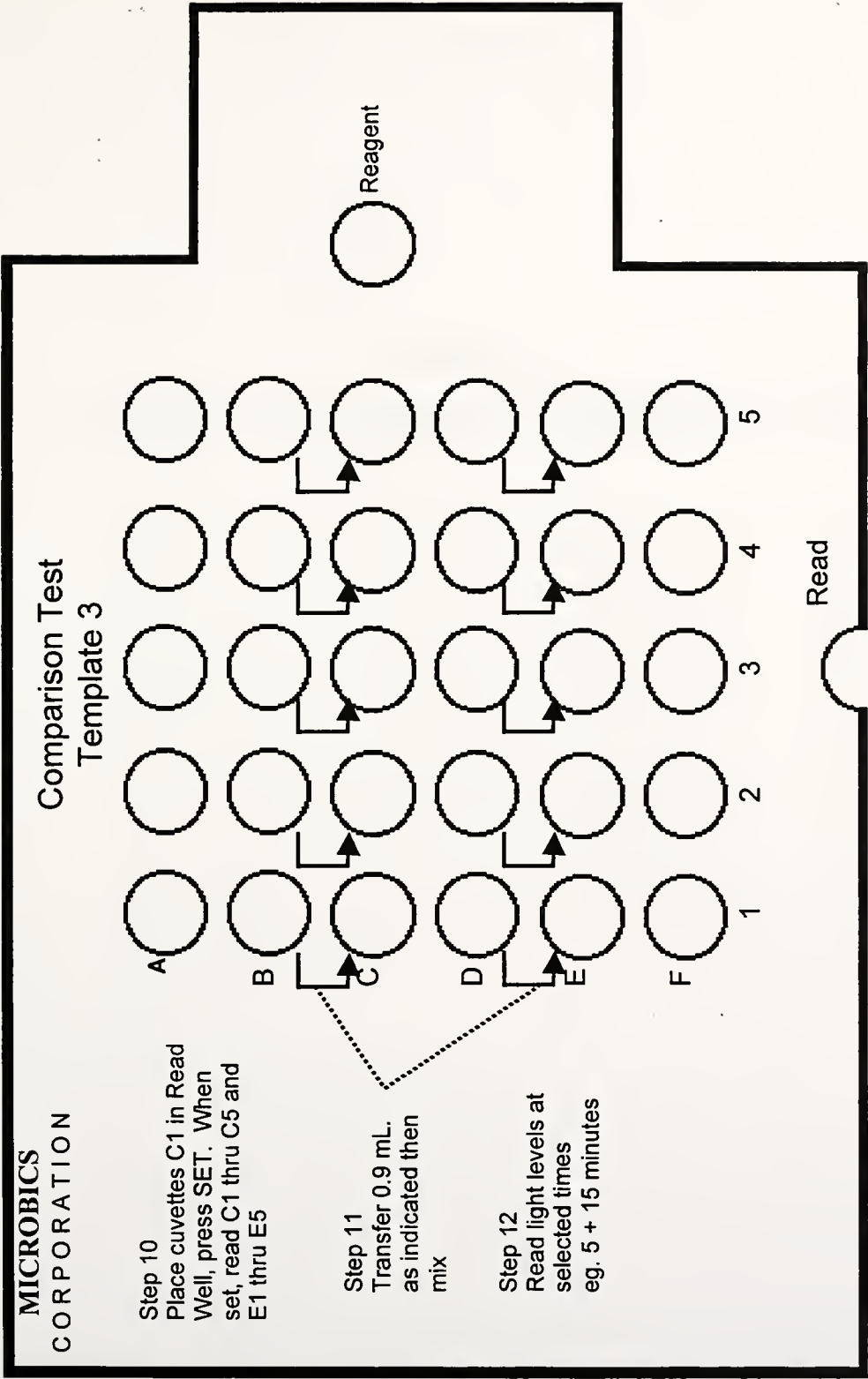


Figure 49 - Microtox™ 90% Comparison Test, Template 1

APPENDIX B

ION CHROMATOGRAPHY AND OPERATION OF THE GP40

(Dionex Corporation, 1983; Dionex Product Selection Guide, 1997-98)

1. Introduction to ion chromatography

Ion Chromatography is the merging of two major areas of development, chromatography and ion exchange. Therefore, it possesses all the HPLC features, but adding something special, for instance, sometimes organic solvent will harm the columns, suppressor reduces the background noise. As you may imagine, on the other hand, most of the operation is same as the regular HPLC. This brief manual will cover the IC and please refer to other chromatography reading materials for a complete coverage.

The IC can be used on the following analysis: chemicals, drinking water, waste water, high-purity water, pesticides, fertilizers, fermentation broths, and protein hydrolysate, etc.

2. Structure of the GP40

The GP40 consists of five major components: solvent delivery pump, conductivity detector, (anion and cation) column, suppressor and injector (or autosampler).

The solvent delivery pump is similar to other HPLC pumps, however, it is designed to be acid/base resistant, which is suitable for the solvent delivery for ion chromatography. The GP40 pump can deliver four different solvents with gradient.

This detector is the most commonly used conductivity detector for IC, through UV and other detectors are still usable.

Cation or anion analytical column can be used depending on what type of ions that are to be analyzed.

The suppressor is a powered self-regenerated-suppressor and is designed specifically for cation or anion.

The injector is similar to other HPLC Rynedyn injectors, however, it can be controlled by the solvent pump CPU, making injections by using compressed air.

3. Operation procedures

i. Prepare your column

Choose right column for anions and cations.

Making sure that the column is connected.

ii. Prepare your eluent

The Standard Method and IC manual have the most commonly used eluent for anion and cation analysis. The eluent must be made up from nanopure or equivalent high quality water.

Filtration must be done for the sake of lengthening the lifetime of the pump delivery system. Make sure you have the right eluent before turning on the pump, especially when using the eluent made

from previous users. The most commonly used eluent for anion analysis is 0.1 mM NaHCO_3 and 0.1 mM Na_2CO_3 .

Check the eluent and d-water for possible bugs grown therein, especially during the summer months, if the sample is not made fresh.

- iii. Prepare your samples
For both qualitative and quantitative analysis, especially those samples from non particle-free sources, must be filtered. An estimation of qualitative samples should be made for their concentration to ensure that the sample can be observed on the chromatogram with reasonable height for peaks. Whereas for quantitative analysis, a estimation for the linear range should be estimated first.
- iv. Check the suppressor
Depending what ions that are to be analyzed, users have to check the suppressor and make sure the suppressor is matched with the ion and the column, before turning on the conductivity detector.
- v. Turn on the IC
Turn on the nitrogen gas and adjust the pressure to about 5 psi, then turn on the eluent pressure switch before turning on the power to the solvent delivery pump. Make sure that the pump is off, then prime the pump. Check if there is any leaking with your column connection. Match the type of ion and type of suppressor and finally turn on the conductivity detector. Turn on the SRS current to 100 mA. When the detector is stabilized, (ca. 10 min), one may start the injection.
- vi. Prime the pump
Move the valve switch 90 degree clockwise. From the direct control menu dial the flow rate to 0 ml/min. For a fresh priming, some iso-propyl alcohol can be used during the priming by pressing it in, but remember to draw it out. When no more bubbles can be found, close the priming valve by turning the valve switch back, counterclockwise 90 degrees (toward to the user).
- vii. Sample analysis
Currently, no autosampler is available for the sample analysis, i.e., all the analyses must be manually injected, monitored by the integrator and quantified by the response curve. The criterion of the chromatogram are the same as for other HPLCs.
- viii. Shut down the IC
Turning down the IC is the reverse of turning it on. Remember to turn the SRS current to 0 mA if the main power remains on during the daily idle.

4. Method Development

Most common applications use isocratic methods for the analysis. Quite a few ions can be found in the Standard Methods or IC manual. Some special attention should be paid to the organic samples but with inorganic anion, to smoothly elute the organic ions, however, some miscible organic solvents have to be introduced.

An AS4A anion column is the default column for anion analysis.

Either pressure or flow rate can be used for the monitoring of the conditions, but the former is more commonly used.

Turn on the offset of the detector then record the analysis.

5. Notes to the operator

- i. Users must wash the column carefully and adequately after his/her using, as most of the time everyone in the area shares the same column
- ii. User must turn the SRS current to off when the detector has zero flow, or not in use. Failure to do so and the CD20 will be damaged permanently!

6. Data interpretation

- i. Qualitative analysis
With current set up, qualitative analysis with sub-ppm concentrations should be easy for detection for most of ions. Thus, the sample concentration should be adjusted accordingly. The loop size is 25 μ l, therefore, a 100 μ l or large syringe should be sufficient for most of applications.
- ii. Quantitative analysis
The detector ranges 0.01 μ s to 3000 μ s. ca. 10^6 for the full scale. So long as the samples do not have too big of a dynamic range, the linearity should be good. A response curve is needed for the quantitative analysis.
- iii. Data verification
The AA and ICP can be used for cation analysis also. If any suspicion is held, verification can be done with the other equipment.

7. Trouble shooting

- i. Zero pressure error
Quite often this error is due to the non-closed valves, or loose column connection.
- ii. Too high pressure error
Blocked guard column, column dirty or even damaged column might cause this.
- iii. Prime problem

Hard to get continuous pumping is the indicator of a priming problem. It is possibly caused by air bubbles in the line, zero pressure of the gas tank, or a closed solvent line.

- iv. Fluctuation of the baseline
 - a. bad suppressor
 - b. dirty column

APPENDIX C
INDUCTIVELY COUPLED PLASMA SPECTROPHOTOMETER TEST
PROTOCOL

1. Introduction

Inductively coupled plasma is commonly abbreviated as ICP. It is a fast pace, high sensitivity (detection limit in ppb level) high resolution (0.018 nm) and high performance for multiple element detection. The operating principle is based on the atomic emission of elements hence, it is much more sensitive than Atomic Absorption, ca. 2-3 order magnitude higher than that of AA. The operation is slightly complex and operation cost is slightly higher, however, if users have the preparation well done in advance, ICP will be a very handy efficient tool for multi-element system analysis. The linearity of the ICP can be ca. 10^6 , i.e. less calibration is needed.

Theoretical aspects and principle of the instrument refer to:

- i. AtomScan 16/25 Spectrophotometers Operator's Manual, Section 1
- ii. Analytical Chemistry Instrument, by Sugur PP

For technical service call 1-800-333-4249. Calling back within 24 hours is the policy of TJA.

2. Structure of the ICP

The major components of the instrument contain:

Power supply mainly provides the energy for the argon plasma.

Argon gas supply provides argon gas to the plasma and the cooling gas to the torch.

Cooling system, remove any excess amount of heat from the system, specifically the RF system.

Sampling System, including the pump and aerosol to feed the sample to the system and autosampler.

Optical Unit distinguishes the signals from different wavelength, amplify the signal and convert optical signal to electrical signal.

Computer Control, uses ThermoSPEC version 5.04/6.10

3. Operation

Instrument Preparation:

- i. Check the argon gas supply, making sure that at least there is more than 200 psi before the operation, if gas tank is used, or check the indicator of the liquid argon tank at the top.
- ii. Check the power supply, making sure that the filter is all right. Vacuum the filter if the filter indicator is on.
- iii. Check the cooling water, making sure that there is a clear, sufficient amount of water for the cooling system.
- iv. Check the ventilation of the ICP, making sure that waste gas can be expelled.

Sample Preparation:

Research samples have to be filtrated before using to ensure the sample solution can be smoothly delivered to the torch. Use any useful information to estimate the concentration of the solution.

An internal standard has to be used for linearity of a quantitative analysis.

Turning on the instrument:

Preparation of ignition:

- i. Turn on the argon gas, then dial to the pressure gauge to 80 psi for routine analysis.
- ii. Turn on the computer and start ThermSPEC software by using AUTOEXEC or STNRUN command under the DOS prompt.
- iii. Insert the sample line into nanopure water container before turning on the plasma.
- iv. When making sure the power supply and cooling water are ready, ignite the torch.

Ignition of the torch:

Go to SETUP under the pull down menu. Using PLSMA CONTROL for ignition of the torch. Press F1 for a fresh ignition. F9 to execute the command. Any fresh ignition has to purge argon for 90 seconds. There is no possibility of shortening this process, however, it can be reduced to a less time when later on reigniting the torch

Instrument:

Check the high voltage of the RF power, check the sample pump rate, check that the cooling water is in circulation and the torch has no overheating symptoms, then the user may start the analysis preparation.

Preparation for Sample Analysis:

Always calibrate the wavelength each day when using the ICP. It is good to do this while waiting for the instrument to warm up, which for a quantitative analyses should be about 30 minutes or more.

Shut Down the System

Shutting down the system is the reverse of turning on. Before shutting down, rinse the sampling system with some di-water for about 5 minutes.

Shut down the power, cooling, and computer but not the power for the main unit unless the ICP is not going to be used for a long time.

4. Method Development

Qualitative Analysis:

- a. Qualitative analysis
Multiquan is a useful method, method development is the same as quantitative analysis. Only four elements are used for the reference. All other elements are assumed to be as proportional as the elements used in the method.
- b. Multiquan Method Development
Under OPERATION select Multiquan Method
Select the Method and key in elements
- c. Multiquan Method Standardization
Select Multiquan Table and key in parameters. Refresh standard if it is needed.
- d. Multiquan Method Analysis
Select Multiquan Analysis. (Please note that the Multiquan Analysis may give as high as 100% error.)

Quantitative Analysis:

- a. Quantitative analysis
In the software, use METHOD under DEVELOPMENT pull down menu. By following the function keys sequentially input elements, wavelength and check interference. Space bar is for selections. Note, quantitative analyses requires all elements standardization for each method, each time while the Multiquan requires one-time standardization for all the samples.
 - Press F1 choose one element at a time.
 - Choose wavelength. Multiple wavelengths are allowed.
 - Double check the IEC, choose the one that has less interference.
 - F2 is used when the ICP is running with a Y sample inlet. Internal standard yttrium or other equivalent is used.
 - F3 Method Info has few things to be changed for most experiments.
 - Input your favorite output format with F4.
 - Press F5 Element Info then F6 to edit the information. Move the cursor to select concentration unit, calibration method, baseline correction method and number of standard for individual element used for the calibration. As the linearity of ICP is fairly good, generally a two data points method should be enough. However, any more accurate method can use multiple data points.
 - Users do not have to change much for the Plasma Info F6. The default is generally enough for most of the application.
 - Function key F8 is used mainly for method protection.
 - F8 Scan can be done either here or exit from the method development.
 - Press F9 to save the method and leave the method development

b. Peak Search/Wavelength Scan

If SCAN has been completed in the method development, skip to next section. Each Method has to be verified by the peak search. When the program meets questionable peaks, it pauses for input from the operator. Press enter to accept the peak, or move the cursor to the desired peak by pressing the arrow keys. Usually the peak with highest intensity will be chosen as the peak for that particular element. Watch out for interference.

c. Standardization

- Press ANALYSIS under OPERATION pull down menu.
- Choose STANDARDIZATION by pressing F3 key.
- Following the direction of the program, input low and high concentration solution.
- Save the standard response.

d. Complete the application method

By applying the same method, numerous samples can be analyzed. One may input sample information etc. without going back to the method development section. Under ANALYSIS pull down the menu:

- Input sample information.
- Input plasma information.
- Input output information. Results can be printed out after each run, if automatic output is selected.
- Complete and save the method by pressing F9.

e. Analyses of Samples

- Press ANALYSIS under OPERATION pull down menu.
- Refresh standards if it is needed.
- RUN sample analysis.

5. Data Treatment and Interpretation:

i. Baseline correction

Use autocorrection. Manual corrections can be done for experienced users only.

ii. Interelement correction (IEC)

IEC choose the wavelength such that no interference elements are present in the sample.

iii. Data analysis

Standardized method will be used as response factor for analyses. Results can be printed out automatically if method is so developed. Routinely, no data storage is needed.

6. Special Notes, Tips and Trouble Shooting:

i. Make sure there is enough argon gas for your samples

ii. Power supply filter quite often has to be cleaned up, otherwise, ignition is impossible. Make sure there is enough cooling water in the jar.

Prepare all the samples before turning on the ICP.

- iii. Minimal sample quantity required may be estimated by the number of elements and the base analysis time, typically 2-6 minutes.
- iv. After analysis, double check the light source, for sample analyses it has to be PLASMA.

APPENDIX D**RANDOM SAMPLING OF A LOT WITHIN A WASTE FOUNDRY SAND****STOCKPILE, INDIANA DEPARTMENT OF TRANSPORTATION****APRIL, 1998**

A lot is defined as a subset of a stockpile of materials which will be used as **embankment material** on an INDOT construction project and which contains approximately 15,300 cubic meters of material. The selection of the sampling locations within a **lot** must be entirely **random** for acceptance of the lot. "Random" means that samples are selected without bias.

The most common method for determining when or where to obtain samples is through the use of a **random number table**. A random number table is a collection of random digits. Random number tables come in many forms: some are short, some are long, some are grouped by pairs of digits, some with as many as 5 digits per group. When using a random number table, the key concern is **bias must be avoided**.

A brief example of a random table is presented at the right. The random numbers are presented in pairs of digits and, for the methods that we consider, can be thought of as two-place decimal fractions. For example, the random number 57 in the table would be 0.57.

When selecting a group of random numbers, one can enter the table at any point (but never at the same point twice) and select the required amount of numbers. The numbers can be selected by columns or rows, by going left or right, up or down, selecting alternate numbers or any other pattern desired.

57	50	75	54	10	74	60	01	27	43
40	50	91	86	58	29	21	78	01	43
89	04	52	53	41	28	37	00	49	97
72	04	68	85	80	73	08	87	32	97
10	58	38	62	63	72	14	09	70	41
03	68	44	84	01	37	84	86	64	51
32	13	15	61	94	36	90	18	02	05
25	12	21	93	33	81	51	85	95	05
63	67	91	60	64	35	37	94	27	95
92	11	68	79	57	35	53	16	65	03
88	10	84	92	96	89	14	93	58	13
26	75	45	79	89	99	20	25	87	68
10	74	60	01	27	43	01	92	70	67
58	29	21	78	01	43	06	24	18	12

Table 12: Random Number Table

Random sampling ensures that each portion of a lot has the same chance of being selected for the sample. **Stratified random sampling** additionally involves the selection of two or more defined parts of a given lot. **Stratified sampling** is used to ensure that the samples are obtained from throughout the lot, and are not concentrated in one portion or section of the lot.

Initially, the volume and portion of a waste foundry sand stockpile that will be used in the embankment needs to be identified. This portion of the stockpile will then be divided into lots not to exceed approximately 15,300 cubic meters per lot, followed by stratified sampling of the lots.

The figure at the right illustrates the basic principle of **stratified sampling**. The lot is then stratified into **sublots** equal to the sample size (as per the *Acceptance Criteria for Waste Ferrous Foundry Sand Reuse Based on the Microtox Bioassay Test*, the sample size, n , is 5). One sample is then randomly selected from within each **sublot**. This ensures that each portion of the lot has the same chance of being selected while, at the same time, ensuring that the sampling is spread out over the entire lot.

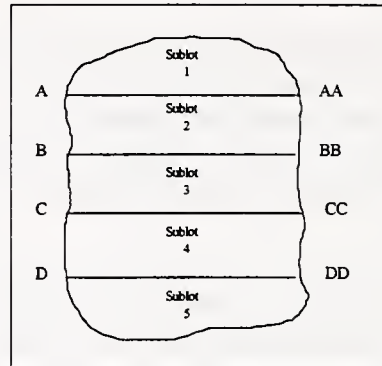


Figure 50 - Stratified Sampling of Sublots

An **example** will help illustrate the use of random number tables and stratified sampling. Suppose that the subplot boundaries have been identified, the location of the sample within each subplot must be determined. To accomplish this, the location must be randomized in the longitudinal (X) as well as the transverse (Y) direction.

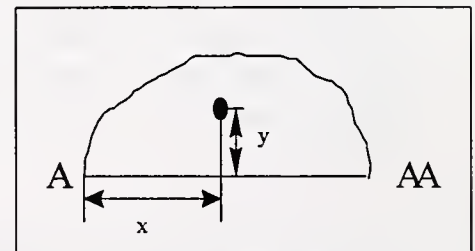


Figure 51 - Sublot Sample Location

The example **random number table** can be used to determine both the transverse and longitudinal locations for the samples. Two sets (columns, rows, etc.) of the numbers are selected, one for the transverse position, the other for the longitudinal position. One more set of random numbers (column or rows, etc.) is needed for the vertical position (Z), or depth, of the sample in the subplot.

A set of 5 random numbers for the longitudinal (X) position, 5 random numbers for the transverse (Y) position and 5 random numbers for the vertical (Z) position may be chosen by using the second block of numbers from the example random number table.

The X, Y and Z random numbers are multiplied by the subplot length, width and vertical depth (Z=3 meters), respectively, as shown in the example below:

74	60	01	27	43	X
29	21	78	01	43	Y
28	37	00	49	97	
73	08	87	32	97	
72	14	09	70	41	Z

Table 13: Random Number Table Example

Sublot #1 (Width = 8.2m, Length = 14.5 m)

Coordinate X = $0.74 \times 14.5 = 10.73\text{m}$.

Coordinate Y = $0.29 \times 8.2 = 2.38\text{m}$.

Coordinate Z = $0.72 \times 3.0 = 2.16\text{m}$.

Sublot #2 (Width = 12.6m , Length = 23.8m)

Coordinate X = $0.60 \times 12.6 = 7.6\text{m}$.

Coordinate Y = $0.21 \times 23.8 = 5.0\text{m}$.

Coordinate Z = $0.14 \times 3.0 = 0.4\text{m}$.

The longitudinal distance (X) is measured from the selected point (A, B, C, or D) and the companion transverse distance (Y) is measured from the point (X distance from A for example) into the subplot. The vertical (Z) distance is measured downward to locate the point (depth) where the sample is going to be taken.

The five samples from each lot will then be evaluated per the *Acceptance Criteria for Waste Ferrous Foundry Sand Reuse Based on the Microtox Bioassay Test* for acceptance or rejection of the lot (stockpile) for reuse as embankment material.

The above mentioned random sampling is for acceptance of a lot within a stockpile. There will be another test, verification test, when the accepted lot is delivered to the job site. One verification test will be taken for every lot ($15,300 \text{ m}^3$) delivered to the job site. If the verification test fails, ($\bar{x} > \bar{x}_A = 12.0$, providing that the variances are homogeneous) another final verification test will be randomly selected and tested. If the final verification test also fails then the lot may be rejected and replaced at the discretion of the Engineer.

APPENDIX E
ACCEPTANCE CRITERIA FOR WASTE FOUNDRY SAND REUSE
BASED ON THE MICROTOX™ BIOASSAY TEST, INDIANA
DEPARTMENT OF TRANSPORTATION
APRIL, 1998

INTRODUCTION

Acceptance Criteria, in this report, is the procedure to determine the acceptance or rejection of a Waste Foundry Sand (WFS) source, from a **ferrous** foundry, for use by the Indiana Department of Transportation (INDOT) in roadway construction, which conforms to Indiana Administrative Code (IAC), 329 IAC 2, for a Type III or Type IV restricted waste which satisfies applicable siting criteria. If a source changes, the Acceptance Criteria must be reapplied.

Basically, there are three approaches to determine the acceptance of a WFS source:

- 1) Accepting the WFS source without any inspection.
- 2) Inspection of the entire WFS source, (100 percent inspection).
- 3) Acceptance Sampling.

Acceptance Sampling lies somewhere between no inspection and 100 percent source inspection. Random sampling is essential for the acceptance sampling. Therefore, the observations (tests) should be representative of the material in the particular WFS source.

Sample size determination for Acceptance Sampling is based on the initial assumptions that:

- Observations are normally distributed.
- Observations are independent.
- Observations are identically distributed with a common variances, σ^2 , i.e. the variances for each normally distributed group are homogeneous (statistically equal).

DERIVATION OF ACCEPTANCE SAMPLING PLAN

Derivation of Acceptance Sampling Plan for Waste Foundry Sands from **ferrous** foundries when the following are Specified :

- The mean of homogeneous populations (WFS source) are considered acceptable, μ_A .
- The mean of homogeneous populations (WFS source) are considered unacceptable, μ_R .
- Type I error, α , probability of rejecting the true mean.
- Type II error, β , probability of accepting the wrong mean.
- Standard Error of Population (WFS source), σ , is a known constant.

The task in designing the Acceptance Sampling Plan is to determine the following:

- Sample size, **n**, the number of test materials (samples) taken randomly throughout the WFS source , and
- $\bar{x}_A$ (*the acceptance limit*)

With respect to Figure 1, we can write the following equations:

$$\bar{x}_A = \mu_A + Z_\alpha \sigma_{\bar{x}} \quad (1)$$

$$\bar{x}_A = \mu_R - Z_\beta \sigma_{\bar{x}} \quad (2)$$

Since the left sides of eq.(1) and eq.(2) are equal, and substituting we then have:

$$n = \frac{[Z_\alpha + Z_\beta]^2}{[\mu_A - \mu_R]^2} \sigma^2 \quad (3)$$

Round **n** to the next high integer number and then compute the acceptance limit from:

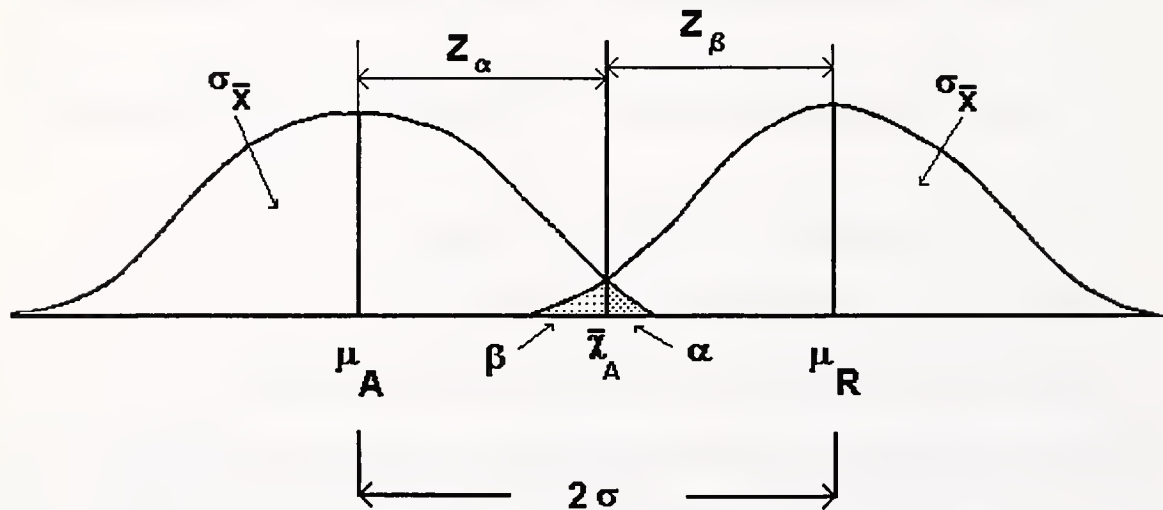
$$\bar{x}_A = \mu_A + Z_\alpha \left(\frac{\sigma}{\sqrt{n}} \right) \quad (4)$$

Decision Rule:

- If $\bar{x} \leq x_A$ then accept the WFS source.
- If $\bar{x} > x_A$ then reject the WFS source.

Where: $\bar{x}$ = The mean of the samples from a particular WFS source.

Figure 52 - Acceptance Criteria Elements



Where:

μ_A = The mean for populations (WFS source) are considered acceptable.

μ_R = The mean for populations (WFS source) are considered unacceptable.

α = Type I error.

β = Type II Error.

$\bar{x}_A$ = The Acceptance Limit (Critical Value).

σ = The standard deviation of the homogenous populations (WFS source)

$\sigma_{\bar{x}} = \frac{\sigma}{\sqrt{n}}$ = The standard deviation of the mean.

Z_α = The Standard Normal Z value for Type I Error, α .

Z_β = The Standard Normal Z value for Type II Error, β .

General Decision Rules:

- Obtain random samples of size n from the WFS source.
- Compute the standard deviation, s_s , of the sample tests and standard deviation, s_c , of the control tests per Microtox™ protocol for each sample at T5 and T15.
- Check the homogeneity of variances between control tests and sample tests for each sample at T5 and T15.

For each sample and time:

$$\text{if } \left(\frac{s_s}{s_c} \right)^2 \leq 9 \quad (5)$$

when $s_s > s_c$, then the variances are homogeneous between control tests and samples tests or

$$\text{if } \left(\frac{s_c}{s_s} \right)^2 \leq 9 \quad (6)$$

when $s_c > s_s$, then the variances are homogeneous between control tests and sample tests.

Note: If variances between the control tests and sample tests are not homogeneous more samples will need to be collected from the WFS source or the source disallowed.

If variances are homogeneous then compute the pooled variance, v^2 , for each combined sample tests and control tests for each time (T5 and T15) from the formula:

$$v^2 = \frac{(s_c^2 + s_s^2)}{2} \quad (7)$$

Check the homogeneity of variances between v^2 and σ^2 , population variance (determined later):

$$\text{When } v^2 > \sigma^2; \quad \text{if } \left(\frac{v^2}{\sigma^2} \right)^2 \leq 9 \quad (8)$$

$$\text{When } \sigma^2 > v^2; \quad \text{if } \left(\frac{v^2}{\sigma^2} \right)^2 \leq 9 \quad (9)$$

then the variances between the population and combination of control tests and sample tests for each sample and time are homogeneous.

Note: If the variances between the population and the combination of control and sample tests are not homogeneous more samples need to be collected from the WFS source or the source disallowed.

If the variances, v^2 , for each sample and time are homogeneous then compute the corresponding normalized variance, s^2 , for each set of control tests and sample tests for each time, eq. (10), and then after compute the pooled normalized variance, s_p^2 , eq.(11)

$$s^2 = \left(\frac{\sqrt{v^2}}{x_c} 100 \right)^2 \quad (10)$$

$$s_p^2 = \frac{1}{p} \sum_{i=1}^p s_i^2 \quad (11)$$

Where: p = number of samples.

s_i^2 = variance of the i -th sample.

x_c = the mean of control test, from Microtox™ data sheet.

σ^2 = the variance of the population to be estimated later.

Check if the variances s_p^2 and σ^2 are homogeneous:

$$\text{When } s_p^2 > \sigma^2; \quad \text{if } \frac{s_p^2}{\sigma^2} \leq 9 \quad (12)$$

$$\text{When } \sigma^2 > s_p^2; \quad \text{if } \frac{\sigma^2}{s_p^2} \leq 9 \quad (13)$$

then variances are homogeneous.

Compute the normalized mean difference, $\bar{x}$, eq.(14), for each combination of control tests and sample tests for each time. Then, compute the pooled normalized mean difference, $\bar{x}_p$, eq.(15) :

$$\bar{x} = \left(\frac{(x_c + x_s)}{2x_c} 100 \right) \quad (14)$$

$$\bar{x}_p = \frac{1}{p} \sum_{i=1}^p \bar{x}_i \quad (15)$$

Where:

p = number of samples.

x_c = the mean of sample test, from microtoxTM data sheet.

$\bar{x}_i$ = the normalized mean difference of the i-th sample.

Decision Rule:

- if $\bar{x}_p \leq \bar{x}_A$ Then accept the WFS source.
- if $\bar{x}_p > \bar{x}_A$ Then reject the WFS source.

Where: $\bar{x}_A$ = The critical value which will be defined later.

SPECIFICATION

- (1) Obtain 5 random samples from the WFS source. For each sample (1 through 5) run the microtoxTM test to obtain control tests mean, $\bar{x}_c$, and sample tests mean, $\bar{x}_s$, for each sample and time.
- (2) Compute the standard deviation, s_c , for control tests and s_s , for sample tests for each sample (1 through 5) and time.
- (3) For each sample and time check to see if the variances between control tests and sample tests are homogenous , (eq.(5) or eq.(6)).
- (4) Compute the pooled variance, v^2 , for each pooled sample tests and control tests for each time (T5 and T15), eq.(7).
- (5) Verify that the variances between the population, σ^2 , and combination of control tests and sample tests, v^2 , are homogeneous, (eq.(8) or eq.(9)).
- (6) Compute the normalized variance, s^2 , for each set of control tests and sample tests for each time, eq.(10).
- (7) Compute the pooled normalized variance , s_p^2 , across samples, eq.(11).
- (8) Check if the pooled normalized variance, s_p^2 , and the population variance, σ^2 , are homogeneous, (eq.(12) or eq.(13)).
- (9) Compute the normalized mean difference, $(\bar{x})$, between the mean of control tests and sample tests, eq.(14).
- (10) Compute the pooled normalized mean differences across samples, $\bar{x}_p$, eq.(15).
- (11) If $\bar{x}_p \leq \bar{x}_A = 12.0$ (for T5 and T15) then accept the WFS source.
 If $\bar{x}_p > \bar{x}_A = 12.0$ (for T5 or T15) then reject the WFS source.

Note: When verifying homogeneity of variances against σ^2 , if σ^2 is larger it automatically indicates homogeneity of variances. If this frequently happens σ^2 should be verified.

Table 14: Example Microtox™ Test Data (Acceptance of WFS Source)

	Sample 1		Sample 2		Sample 3		Sample 4		Sample 5	
	T5 ⁽²⁾	T15	T5	T15	T5	T15	T5	T15	T5	T15
Control Test <i>Mean, x_c</i> ⁽¹⁾	92.35	89.08	99.08	84.16	92.89	81.15	103.50	95.79	101.89	88.09
<i>Std. s_c</i>	2.07	0.93	1.64	1.73	2.15	3.31	2.01	2.12	1.38	1.06
Sample Test <i>Mean x_s</i> ⁽¹⁾	89.23	102.62	97.50	92.77	89.65	85.76	100.49	101.10	100.47	96.27
<i>Std. s_s</i>	2.28	2.88	3.79	5.23	2.31	2.80	4.16	4.53	3.27	1.73
Normalized Mean Difference, $\bar{x}$ ⁽¹⁾	3.4	-15.2	1.6	-10.2	3.5	-5.7	2.9	-5.5	1.4	-9.73
Pooled Normalized Std. s ⁽³⁾ ($t_{8,0.025}=2.306$)	5.4/2.306 =2.4	2.2	2.7	3.1	2.4	3.8	3.0	3.5	2.3	1.6

⁽¹⁾ Data taken from Microtox™ test results.

⁽²⁾ T5 and T15 are Microtox™ test results at 5 minutes and 15 minutes, respectively.

⁽³⁾ Data taken from Microtox™ test results divided by 2.306. ($(t_{8,0.025}=2.306)$ = Standard Student-t-Distribution value for 8 degrees of freedom and $\alpha=0.025$).

Specification Rules:

- (1) 5 random samples were obtained from the WFS source. The microtoxTM test was run to obtain data shown in Table 1.
- (2) For Sample 1 at T5, for instance, we calculate $s_c=2.07$ for control tests; $s_s=2.28$ for sample tests.
- (3) Check for homogeneity of variances between control tests and sample tests using eq.(5) as $s_s > s_c$:

$(2.28/2.07)^2 = 1.2 < 9.0$ therefore, there is homogeneity of variances between control tests and sample tests.

Homogeneity of variances between control tests and sample tests would similarly be found to be true for all samples and for all times.

- (4) The combined variance, v^2 , for the T5 test using eq.(7) is, $v^2 = 4.74$, and The combined variance, v^2 , for the T15 test using eq.(7) is, $v^2 = 4.56$.
- (5) Verify that the variances between the population, σ^2 , and combination of control tests and samples, v^2 , are homogeneous, using eq.(8), as : $\sigma^2 > v^2$:

$(\sigma^2 / v^2) = (9.0/4.74) < 9.0$, therefore the variances are homogeneous for the T5 test..
 $(\sigma^2 / v^2) = (9.0/4.56) < 9.0$, therefore the variances are homogeneous for the T15 test.

- (6) The normalized variance, s^2 , for the T5 test using eq.(10) is, $s^2 = 2.4^2$, and the normalized variance, s^2 , for the T15 test is, $s^2 = 2.2^2$.

- (7) The pooled normalized variances, s_p^2 , across samples are, from eq.(11):

For T5: $s_p^2 = (2.4^2 + 2.7^2 + 2.4^2 + 3.0^2 + 2.3^2) / 5 = 6.25$

For T15: $s_p^2 = (2.2^2 + 3.1^2 + 3.8^2 + 3.5^2 + 1.6^2) / 5 = 8.74$

- (8) Check if the variances, s_p^2 , and σ^2 are homogeneous using eq.(13) as $\sigma^2 > s_p^2$:
 For T5: $(9.0/6.25) < 9$ then variances are homogeneous .

For T15: $(9.0/8.74) < 9$ then variances are homogeneous .

- (9) Compute the normalized mean difference, $\bar{x}$, between the mean control tests and sample tests using eq.(14):

For T5: $\bar{x} = 3.4$

For T15: $\bar{x} = -15.2$

- (10) Compute the pooled normalized mean difference across samples for each time (T5 and T15) are, eq.(15):

For T=5 : $\bar{x}_p = 2.5$

For T=15: $\bar{x}_p = -9.3$

- (11) Note that ($\bar{x}_p = 2.5 < \bar{x}_A = 12.0$) for the T5 test results and ($\bar{x}_p = -9.3 < \bar{x}_A = 12.0$) for the T15 test results, therefore accept the WFS source.

Table 15: Example Microtox™ Test Data (Rejection of WFS Source)

		Sample 1		Sample 2		Sample 3		Sample 4		Sample 5	
		T5 ⁽²⁾	T15	T5	T15	T5	T15	T5	T15	T5	T15
Control Test	Mean, x_c Std. s_c	94.41 1.9	82.02 1.7	103.53 2.7	90.05 1.8	100.21 2.9	90.60 1.8	104.81 3.7	92.59 3.1	99.56 2.1	86.13 1.3
Sample Test	Mean x_s Std. s_s	76.13 1.8	97.24 2.19	67.62 1.4	60.36 1.0	72.55 5.1	55.01 3.9	76.74 2.1	71.25 1.73	89.42 1.4	82.71 2.1
Normalized Mean Difference, $\bar{x}_i$ ⁽¹⁾		19.4	18.0	34.7	33.0	27.6	39.3	26.8	23.0	10.2	4.0
Pooled Normalized Std. s ⁽³⁾ ($t_{8,0.025}=2.306$)		4.5/2.306 =2.0	2.3	2.0	1.6	4.0	3.2	2.7	2.6	1.8	1.9

⁽¹⁾ Data taken from Microtox™ test results.

⁽²⁾ T5 and T15 are Microtox™ test results at 5 minutes and 15 minutes, respectively.

⁽³⁾ Data taken from Microtox™ test results divided by 2.306. ($t_{8,0.025}=2.306$) = Standard Student-t-Distribution value for 8 degrees of freedom and $\alpha=0.025$).

Specification Rules:

- (1) 5 random samples were obtained from the WFS source. The microtoxTM test was run to obtain data shown in Table 2.
- (2) For Sample 1 at T5, for instance, we calculate $s_c=1.9$ for control tests; $s_s=1.8$ for sample tests .
- (3) Check for homogeneity of variances between control tests and sample tests using eq.(6) as $s_c > s_s$:

$(1.9/1.8)^2 = 1.2 < 9.0$ therefore, there is homogeneity of variances between control tests and sample tests.

Homogeneity of variances between control tests and sample tests would similarly be found to be true for all samples and for all times.

- (4) The combined variance, v^2 , for T5 test using eq.(7) is, $v^2 = 3.43$, and the combined variance, v^2 , for the T15 test is, $v^2 = 3.85$.
- (5) Verify that the variances between the population, σ^2 , and combination of control tests and samples, v^2 , are homogeneous using eq.(9) as : $\sigma^2 > v^2$:

$(\sigma^2 / v^2) = (9.0/3.43) < 9.0$, therefore the variances are homogeneous for the T5 test..

$(\sigma^2 / v^2) = (9.0/3.85) < 9.0$, therefore the variances are homogeneous for the T15 test.

- (6) The normalized variance, s^2 , for the T5 test using eq.(10) is , $s^2 = 2.0^2$, and the normalized variance, s^2 , for the T15 test is , $s^2 = 2.3^2$.

- (7) The pooled normalized variances, s_p^2 , across samples are, eq.(10):

For T5: $s_p^2 = 6.76$

For T15: $s_p^2 = 5.76$

- (8) Check if the variances, s_p^2 , and σ^2 are homogeneous, eq.(11).
 $(9.0/6.76) < 9$) then variances are homogeneous for T5 test.
 $(9.0/5.76) < 9$) then variances are homogeneous for T15 test.

- (9) The normalized mean difference, $\bar{x}$, between the mean control tests and sample tests, eq.(12).

For T5: $\bar{x} = 19.4$

For T15: $\bar{x} = 18.0$

- (10) The pooled normalized mean difference across samples for each time (T5 and T15) are, eq.(13):

For T=5 : $\bar{x}_p = 23.74$

For T=15: $\bar{x}_p = 23.46$

- (11) ($\bar{x}_p = 23.74 > \bar{x}_A = 12.0$) then reject the WFS source using T5 test results.

($\bar{x}_p = 23.46 > \bar{x}_A = 12.0$) then reject the WFS source using T15 test results.

Table 16: Example Microtox™ Test Data (Rejection of WFS Source, Non-Homogeneous Variances)

		Sample 1		Sample 2		Sample 3		Sample 4		Sample 5	
		T5 ⁽²⁾	T15	T5	T15	T5	T15	T5	T15	T5	T15
Control Test	<i>Mean, x_c</i> ⁽¹⁾	94.41	82.02	103.53	90.05	100.21	90.60	104.81	92.59	99.56	86.13
	<i>Std. s_c</i>	10.1	9.7	12.7	13.5	12.9	10.8	13.7	13.1	12.1	11.3
Sample Test	<i>Mean x_s</i> ⁽¹⁾	85.32	77.24	97.62	80.36	92.55	79.01	9.74	81.25	89.42	70.71
	<i>Std. s_s</i>	8.8	12.2	11.4	11.0	9.	13.9	12.1	11.73	11.4	12.1
Normalized Mean Difference, $\bar{x}_i$ ⁽¹⁾		9.4	8.0	8.7	13.0	7.6	9.3	9.7	13.0	10.2	8.0
Pooled Normalized Std. s_i ⁽³⁾ ($t_{8,0.025}=2.306$)		20.1/2.306 8.7	12.8	12.0	11.6	14.0	13.2	12.7	12.6	11.8	11.9

(1) Data taken from Microtox™ test results.

(2) T5 and T15 are Microtox™ test results at 5 minutes and 15 minutes, respectively.

(3) Data taken from Microtox™ test results divided by 2.306. ($(t_{8,0.025}=2.306)$ = Standard Student-t-Distribution value for degree of freedom=8 and $\alpha=0.025$).

Specification Rules:

- (1) 5 random samples were obtained from WFS source. The Microtox™ test was run to obtain data shown in table 1.
- (2) For Sample 1 at T5, for instance, we calculate $s_c=10.1$ for control tests; $s_s=8.8$ for sample tests at T5.
- (3) Check for homogeneity of variances between control tests and sample tests using eq.(5):

$(10.1/8.8)^2 = 1.2 < 9.0$ therefore, there is homogeneity of variances between control tests and sample tests.

Homogeneity of variances between control tests and sample tests would similarly be found to be true for all samples and for all times.

- (4) The pooled variance, v^2 , for T5 test is, $v^2 = 89.73$, and
The pooled variance, v^2 , for T15 test is, $v^2 = 121.47$, eq.(7).
- (5) Verify that the variances between the population, σ^2 , and combination of control tests and samples, v^2 , are homogeneous, eq.(8):

$(v^2/\sigma^2) = ((89.73/9.0) > 9.0)$ therefore the variances are homogeneous for T5 test..

$(v^2/\sigma^2) = (121.47/9.0) > 9.0$ therefore the variances are homogeneous for T15 test.

- (6) The normalized variance, s^2 , for T5 test is, $s^2 = 8.7^2 = 70.69$, and
The normalized variance, s^2 , for T15 test is, $s^2 = 12.8^2 = 163.84$, eq.(9).

- (7) Pooled Normalized Variances, s_p^2 , across samples are, eq.(10):

For T5: $s_p^2 = 144.0$, and

For T15: $s_p^2 = 153.76$

- (8) Check if the variances, s_p^2 , and σ^2 are homogeneous, eq.(11).
 $(144.0/9.0) > 9.0$ then variances are not homogeneous for T5 test.
 $(153.76/9.0) > 9.0$ then variances are not homogeneous for T15 test.

- (1) The normalized mean difference, $\bar{x}$, between the mean control tests and sample tests, eq.(12).

For T5: $\bar{x} = 9.4$

For T15: $\bar{x} = 8.0$

- (2) The pooled normalized mean difference across samples for each time (T5 and T15) are, eq.(13):

For T=5 : $\bar{x}_p = 9.12$, and

For T=15: $\bar{x}_p = 10.3$

- (3) ($\bar{x}_p = 9.12 < \bar{x}_A = 12.0$) then accept the stockpile using T5 test results.
 ($\bar{x}_p = 10.3 < \bar{x}_A = 12.0$) then accept the stock pile using T15 test results.

However, due to non-homogeneous variances found in the item (8), the WFS source had to be rejected.

SPECIFICATION DEVELOPMENT CRITERIA

Using the virgin sand (natural construction and foundry sands) Microtox™ test results taken from the thirteen random sand sources (Bastian, 1996), the following required tests were performed for all the data.

(1) Normality Check:

The normality check was performed by location, time, control and sample. Each group (control and sample for each time) contained five data points. The test results, using Wilk-Shapiro Normality Test Method indicated that the data was approximately normal.

(2) Homogeneity of variances test:

The homogeneity of variances test for all the groups, each containing five observations, using Bartlett's test of equal variances, indicated that the variances are statistically equal (homogeneous).

(3) Independency of Observations Check:

All the observations are collected randomly and independently.

The next step was to compute the mean of the acceptable WFS source (μ_A) and corresponding standard deviation (σ). The data obtained from the thirteen independent sand sources was used and the results are listed in Table 1.

The Statistical software "STATISTIX FOR WINDOWS" was used for the above mentioned tests.

Assumptions:

- $\mu_R - \mu_A = 1.5 \sigma$
- $\alpha = 0.05$
- $\beta = 0.05$

Table 17: Virgin Sand Microtox™ Comparison Data

VIRGIN SAND LOCATION	NORMALIZED MEAN DIFFERENCES BETWEEN CONTROL TESTS AND SAMPLE TESTS ⁽¹⁾ $\bar{x}$		POOLED NORMALIZED STANDARD DEVIATION OF CONTROL TESTS AND SAMPLE TESTS $s^{(3)}$	
	T5 ⁽²⁾ (5 Minute Test)	T15 (15 Minutes Test)	T5 (5 Minutes Test)	T15 (15 Minutes Test)
1	-3.1	-2.0	8.3/2.306=3.6	10.2/2.306=4.4
2	25.1	24.8	1.7	2.7
3	9.1	8.4	1.6	1.8
4	11.5	7.8	1.9	2.6
5	14.4	14.2	2.3	2.3
6	-3.8	0.4	1.3	1.2
7	14.2	12.0	2.7	3.2
8	12.8	13.0	2.3	3.1
9	8.5	6.9	2.7	3.2
10	2.5	5.7	2.0	2.4
11	6.0	4.3	3.6	4.1
12	7.1	9.8	3.4	3.8
13	12.7	13.7	2.3	2.8
MEAN ($\bar{x}_p$)	9.0	9.2		

⁽¹⁾ = The data was obtained from Microtox™ test results.

⁽²⁾ = T5 and T15 are Microtox™ test results at 5 minutes and 15 minutes, respectively.

⁽³⁾ = Data taken from Microtox™ test results /2.306 ($t_{8,0025} = 2.306$ for standard t-Student distribution value for 8 degree of freedom and $\alpha = 0.025$).

The normalized standard deviation , σ , for the population can then be obtain as:

$$\sigma \cong \sqrt{\frac{\sum_{i=1}^p (n_i-1)s_i^2}{\sum_{i=1}^p (n_i-1)}} \quad i=1,2,\dots,p \quad (14)$$

or

when number of observations are all equal to n, the above formula can be simplified as:

$$\sigma = \frac{1}{p} \sqrt{\sum_{i=1}^p s_i^2} \quad i=1,2,3,\dots,p \quad (15)$$

Where:

p = the number of virgin sand locations, in this case 13 locations were used.

s = The pooled normalized standard deviation of control tests and sample tests for a sample.

Since, for each virgin sand location five control tests and five sample tests were performed, using eq.(15), the pooled normalized standard deviation for the population (WFS source) is computed as:

$$\text{For T5: } \sigma = \sqrt{\frac{(3.6^2 + 1.6^2 + \dots + 2.3^2)}{13}} = 2.5 \text{ and}$$

$$\text{For T15: } \sigma = \sqrt{\frac{(4.4^2 + 2.7^2 + \dots + 2.7^2)}{13}} = 3.1$$

The Acceptance Sampling Plan based on the microtoxTM values for virgin sands can be developed for *either T5 and T15 tests* as the normalized mean differences and the normalized pooled standard deviations of T=5 minutes test and T=15 minutes test were found statistically equal. For this reason, we can compute the required pooled normalized standard deviation, σ , and the pooled normalized mean difference, μ_A as follows:

$$\sigma \cong \sqrt{\frac{2.5^2 + 3.1^2}{2}} = 2.8 \cong 3.0, \quad \begin{array}{l} \text{population normalized standard} \\ \text{deviation for T5 and T15.} \end{array}$$

$$\mu_A \cong (9.0 + 9.2)/2 = 9.1, \quad \begin{array}{l} \text{population normalized mean} \\ \text{difference.} \end{array}$$

(Data from Table 4)

Computation of n (samples), and $\bar{x}_A$:

Using eq.(3) and referring to Figure 1, the required number of samples, n , is computed as:

$$n = \frac{(Z_{\alpha=0.05} + Z_{\beta=0.05})^2}{(\mu_A - \mu_R)^2} \sigma^2 = \frac{(1.645 + 1.645)^2}{(1.5\sigma)^2} \sigma^2 = 4.8 \cong 5$$

The control tests and samples tests are normally distributed with common variance, σ^2 , which was estimated as 9.0. The differences between control tests and samples tests are also normally distributed with common variance, $\sigma^2 + \sigma^2 = 2\sigma^2$, and their common standard deviation is equal to $\sqrt{2\sigma^2} = \sqrt{2}\sigma$. We then can calculate the critical difference value, $\bar{x}_A$, between control tests and sample tests referring to Figure 1 and using $\sqrt{2}\sigma$ instead of σ in eq.(4) and setting $n=5$, as:

$$\bar{x}_A = \mu_A + Z_{\alpha=0.05} \sqrt{2} \frac{\sigma}{\sqrt{n}} = 9.1 + 1.645 \sqrt{2} \frac{3.0}{\sqrt{5}} = 12.2 \cong 12.0$$

Where:

$Z_{\alpha=0.05} = 1.645$ is the standard normal Z-value at $\alpha=0.05$ confidence level.

$Z_{\beta=0.05} = 1.645$ is the standard normal Z-value at $\beta=0.05$ confidence level.

APPENDIX F

PROPOSED RECURRING SPECIAL PROVISION FOR

EMBANKMENTS CONSTRUCTED OF SPENT FOUNDRY SAND

AUGUST, 1998

DESCRIPTION: This work shall consist of using spent foundry sand (SFS) as borrow. SFS shall not be used as backfill for MSE walls nor within 0.3 meter of subsurface drain trenches unless otherwise approved. Adherence to the provisions herein does not preclude applicability of local, state or federal regulations and laws.

MATERIALS: SFS is spent foundry sand produced from the ferrous casting industry. These by-products shall be type III or type IV materials per IDEM's restricted waste criteria with metal concentrations less than 80% of the respective maximum allowable type III concentration. Type III SFS with higher metal concentrations may be used provided the foundry indemnify INDOT from liability of clean-up costs which may result from the use of the SFS. SFS shall be in accordance with 203.08 for borrow unless otherwise stated herein. Current production SFS shall not contain baghouse fines, floor sweepings, large slag pieces (> 0.30 m), or tire puncture hazards.

The maximum fines content for SFS shall be limited to 40% (dry unit wt.) unless otherwise approved. Fines shall be defined as that portion of SFS passing the 75µm (#200) sieve.

SFS shall be supplied dry or in a moist condition and transported to the project in a manner that prevents the release of dust and loss of material.

The Contractor shall provide the Engineer with a certification stating the IDEM restricted waste type and that metal concentrations are less than 80% of the respective maximum allowable type III concentration and that the SFS has passed the INDOT Microtox™ bioassay acceptance criteria as described by Indiana Test Method 805-98T. Use of Type III SFS with higher metal concentrations require an executed indemnification clause between INDOT and the foundry. The form of the certification shall be as follows:

SFS SOURCE CERTIFICATION

This is to certify that all spent SFS produced by the _____
 (Foundry) of _____ (Company) located in
 _____ (City), _____ (State), shipped for use on Indiana
 Department of Transportation projects is type _____ (III or IV) material
 according to IDEM's restricted waste criteria with metal concentrations less than 80% of
 the respective maximum allowable type III concentration, or if the metal concentrations
 exceed 80% of the respective maximum allowable type III concentration, but are still
 within the type III criteria, that the foundry has provided indemnification to the Indiana
 Department of Transportation. Furthermore, that the material has passed the Microtox™
 bioassay acceptance criteria as described by Indiana Test Method 805-98T.
 _____ (Foundry) also agrees that any part of the named foundry associated
 with the production of such SFS may be checked at regular intervals by properly
 identified representatives of the Indiana Department of Transportation or a duly assigned
 representative

_____ (Date) _____ (Foundry)
 _____ (Title) _____ (Signature)
 State of _____) SS: County of _____)
 Subscribed and sworn to before me by _____ of the firm of
 _____ this _____ day of _____ 19_____.

_____ Notary Public

My Commission Expires: _____

The Contractor shall, if requested, furnish the Engineer with a copy of the most recent testing results upon which the certification is based. This shall include the following information:

- a. entity performing the test,
- b. date samples were obtained,
- c. date samples tested,
- d. test methods used,
- e. frequency of sampling,
- f. stockpile sampling locations including depths and available historical testing results.

The Department reserves the right to conduct independent quality assurance testing at any time and may reject non-conforming material.

Construction Requirements

SFS not incorporated into the contract through placement, compaction, and encasement within five calendar days will be considered to be in storage. Prior to storing SFS within the contract limits, the Contractor shall have an approved erosion control plan to prevent SFS runoff and erosion. Total SFS in storage shall not exceed 7000 cubic meters and the maximum time in storage shall be 180 calendar days.

Adequate measures shall be taken during construction to control dust. Spraying with water, lime water, bituminous sprays, or other sealing sprays will be considered to be acceptable methods for dust control.

Type III and Type IV SFS shall not be placed as follows:

- (a) Below the seasonal high water table
- (b) Within 30 horizontal meters of a perennial stream/river and lake/reservoir.
- (c) Within 46 horizontal meters of a well, spring, or other ground water source of potable water
- (d) Adjacent to a wetland or other protected environmental resource area.

It will be the Contractor's responsibility to prepare bids for this item by anticipating placement limits and estimating quantities of SFS by referencing to the restrictions set out by (a) through (d).

The placement and compaction of SFS shall be performed in accordance with 203.23 except that unless otherwise approved in writing, the contractor and supplier shall arrange to conduct test strips to determine appropriate compaction methods and moisture control limits. The construction of these test strips will be as directed by the Engineer. Based on the results of the test strips the Engineer will determine appropriate compaction and moisture control criteria.

Nuclear gauges shall not be used to measure moisture or density unless a new calibration curve is made for the SFS and approved by the Engineer.

Encasement material shall be placed concurrently with the SFS. Encasement material shall be placed in accordance with Section 203.09. Areas of SFS adjoining dissimilar materials (excluding encasement) shall be benched to prevent slope failures and control differential settlement.

Encasement shall be soil as specified in 203.09. SFS shall not be used as encasement. SFS shall be covered with a minimum of 0.3 meters of soil. Soil encasement shall be placed and compacted at the same time as the SFS lifts. All cover materials shall be appropriately seeded and vegetated in accordance with 203.09.

METHOD OF MEASUREMENT. SFS and encasement will be measured by the cubic meter.

BASIS OF PAYMENT. SFS embankments will be paid as borrow at the contract unit price per cubic meter placed and compacted.

Payment will be made under:

Pay Item

Pay Unit

Borrow

Cubic Meter

The costs of the construction of test strips, water, lime water, bituminous sprays, or other sealing sprays necessary for dust control, or for moisture content will be included in the cost.

